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# BYPRODUCTS
# FROM
# MILK

BY
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*Gather up the fragments that remain that nothing be lost*
John 6: 12

# PREFACE

There is a widespread and increasing interest in profitable means of utilization of dairy byproducts. Because of their great and unique nutritional value, the most logical method of disposition of these byproducts is, from the standpoint of the general welfare, in food; usually this is also the most gainful method. The use of skim milk, buttermilk and whey in feeding calves, pigs and poultry is warranted to a limited extent by the unique and essential nutritional characteristics of these substances, and this use has been justified to a greater extent by the relatively high cost of other protein feeds. The increasing tendency for dairy farmers to sell whole milk rather than cream and to use greater quantities of protein feeds not derived from milk transfers to a proportional degree to the processors of milk the problems of byproducts disposal. This situation and the likelihood of there being in the future increasingly large surpluses of milk have caused the manufacturers of dairy products to consider more seriously than ever before the marketable products that may be made from milk byproducts and surpluses.

This book has been written with the purpose of collecting in one volume the methods of dairy byproduct utilization that have been available only separately, in Federal and state bulletins, in patents, and in technical journals. Not only have processes in commercial operation been described, but also procedures have been outlined that, though not as yet commercialized, seemed to the authors to offer promise of practical use, at least in principle, even if not precisely as presented. Several products relatively high in fat and made on a small scale only have been described, since they are not widely known and are not main products of the dairy industry.

Such theoretical discussions have been included as seemed necessary for the understanding of the principles on which the manufacturing procedures are based. The references at the end of each chapter do not cover all the literature on each subject, but have been selected as those furnishing the most practical and adequate information on the principles and procedures presented.

The effort has been made to give the information needed by students of dairy science, by research workers, and by persons who will be supervising each process or manufacturing special food products. In some instances the writing is directed to operators of small dairy or other food manufacturing plants, in others to plant managers or engineers. The extent of the treatment of each subject has been largely determined by the amount of information available and by the authors' judgment as to its pertinency.

Temperatures have been given throughout in Fahrenheit degrees, since in the United States the Fahrenheit scale is used generally in industry, with the exception of chemical plants. Centigrade degrees have been given also in a few instances in which it appeared that this might be helpful.

We are grateful to Lucien V. Rogers for his contribution as senior author of Chapter 7 on Bakery Products, to colleagues in this Bureau and in the dairy industry who have made suggestions and have furnished technical information and flowsheets, and to Carol Gard and Veda Spangler who typed the manuscript with painstaking care and helped to avoid or correct many errors.

*Washington, D. C.*
*February 1950*

Earle O. Whittier
Byron H. Webb

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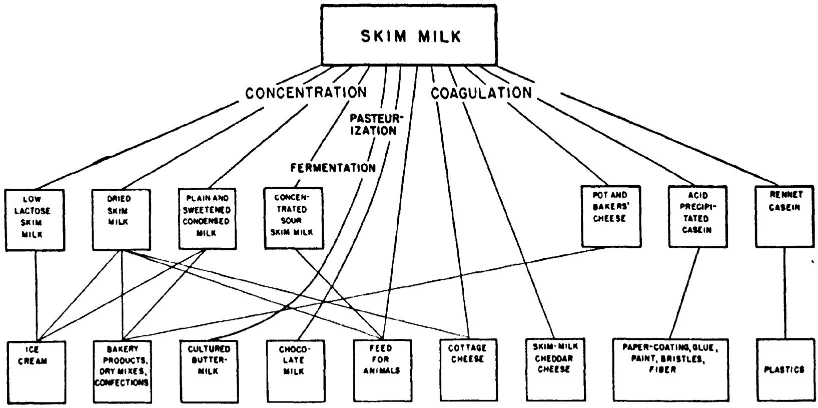


Fig. 1. Products from skim milk.

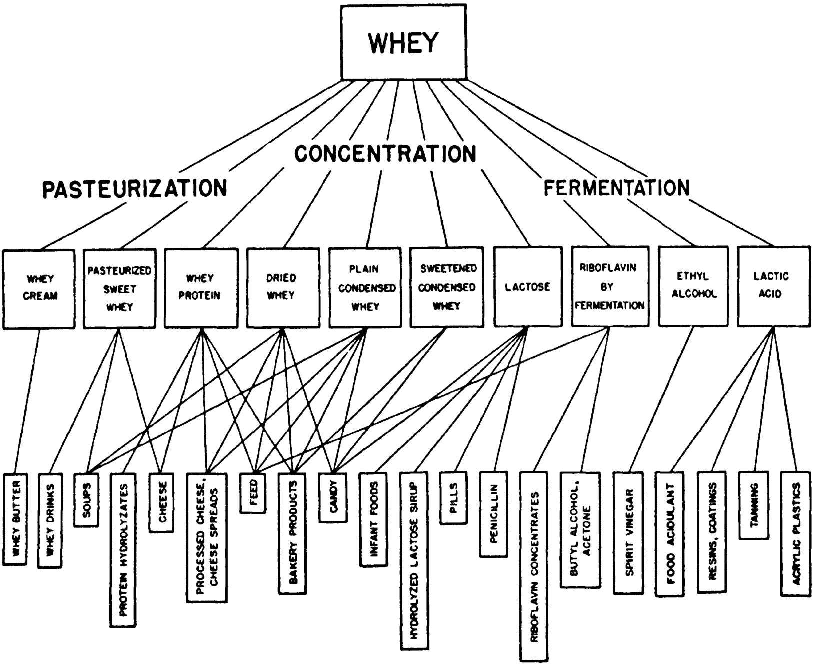


Fig. 2. Products from whey.

# 1

# THE BYPRODUCTS OF MILK

## INTRODUCTION

Skim milk, whey and buttermilk are the byproducts of the dairy manufacturing industry—residues from the manufacture of cream, cheese and butter.

Before discussing the characteristics of dairy byproducts and the methods by which these byproducts may be utilized, it will be profitable to view the gross picture of milk utilization in the United States for 1947. It is not adequate to state merely that, in 1947, approximately 48 percent of the domestic milk supply was utilized as market milk and cream, 27 percent as butter, 10 percent as cheese, 6 percent as evaporated and condensed milk, 6 percent as ice cream, and 1 percent as dried milk. These figures give a total of 98 percent utilization as food, which appears highly satisfactory, the remaining 2 percent being accounted for as milk fed to young animals. But in some of these products only a part of the milk is utilized.

When an attempt is made to account for the solids of milk, it is found that in the products listed above only 63 percent of the solids was utilized. The other 37 percent of the milk solids was in the 52 billion pounds of skim milk, 13 billion pounds of whey and 2 billion pounds of buttermilk that were the byproducts of cream separation, the manufacture of cheese and casein, and the churning of butter. An additional 6 percent of solids from these byproducts was utilized as food. Thus, of the total milk produced, 69 percent of the total milk solids were utilized as food. If this figure is broken down into percentages of each of the three principal nutrients of milk, it is found that, 98 percent fat, 61 percent protein, and 54 percent lactose were utilized in food.

Table 1 indicates the calculated utilization of the principal constituents of milk in food and in concentrated and dried products, which are used mostly for feed for animals. Because typical analyses of somewhat uncertain accuracy were used in making these calculations, the values must be considered only approximately correct. They do, however, indicate with adequate precision the relative quantities of nutrients in the various products. It can be seen also from this table that approximately 37 percent, or 1.5 billion pounds, of our milk protein and approximately 43 percent, or 2.5 billion pounds, of lactose was fed to animals in the form of fluid skim milk, buttermilk and whey, or to some slight extent run to waste as whey.

It is this 4 billion pounds of milk solids that is being utilized at low efficiency, from the standpoint of human nutrition. It has been estimated that, to obtain 1 pound of meat protein, it is necessary to feed an animal approximately 15 pounds of milk protein. More and more emphasis is being placed on the nutritional value of the nonfat portion of milk, because of its unique combination of vitamins, salts and protein. Since milk protein contains all the essential amino acids required by man, it should be rated at least as valuable as meat protein in our nutritional economy. All of these considerations point to the desirability of increased consumption of skim milk as food, and of foods made wholly or in part from skim milk, buttermilk and whey.

TABLE 1. *Utilization of Milk Constituents in Market Products* (1947)
(In million pounds)

| Product | Gross Weight | Total Milk Solids | Fat | Protein | Lactose |
|---|---|---|---|---|---|
| Milk produced on farms | 119,336 | 15,514 | 4,654 | 4,177 | 5,847 |
| Market milk & cream (as milk) | 57,295 | 5,815 | 2,235 | 1,404 | 1,948 |
| Butter | 1,646 | 1,341 | 1,325 | 10 | 7 |
| Whole milk cheese | 1,101 | 694 | 354 | 282 | |
| Cottage cheese | 231 | 53 | 9 | 30 | |
| Plain condensed milk | 175 | 52 | 15 | 13 | 21 |
| Sweetened condensed milk | 247 | 74 | 21 | 19 | 30 |
| Evaporated milk | 3,208 | 844 | 253 | 224 | 318 |
| Concentrated skim milk (food) | 949 | 285 | 2 | 106 | 151 |
| Concentrated skim milk (feed) | 19 | 6 | | 2 | 3 |
| Concentrated buttermilk | 189 | 53 | 3 | 20 | 26 |
| Dried whole milk | 165 | 161 | 44 | 43 | 64 |
| Dried skim milk (food) | 678 | 656 | 7 | 242 | 353 |
| Dried skim milk (feed) | 22 | 21 | | 8 | 12 |
| Dried buttermilk | 45 | 44 | 2 | 16 | 21 |
| Dried whey | 158 | 151 | 1 | 20 | 113 |
| Malted milk | 37 | 18 | 3 | 5 | 8 |
| Ice cream | 2,893 | 665 | 376 | 113 | 153 |
| Chocolate & cultured milks (estimated) | 2,000 | 190 | 2 | 70 | 100 |
| *Totals* | | 11,123 | 4,652 | 2,628 | 3,328 |
| Percentage of that in milk produced on farms | | 72 | 100 | 63 | 57 |

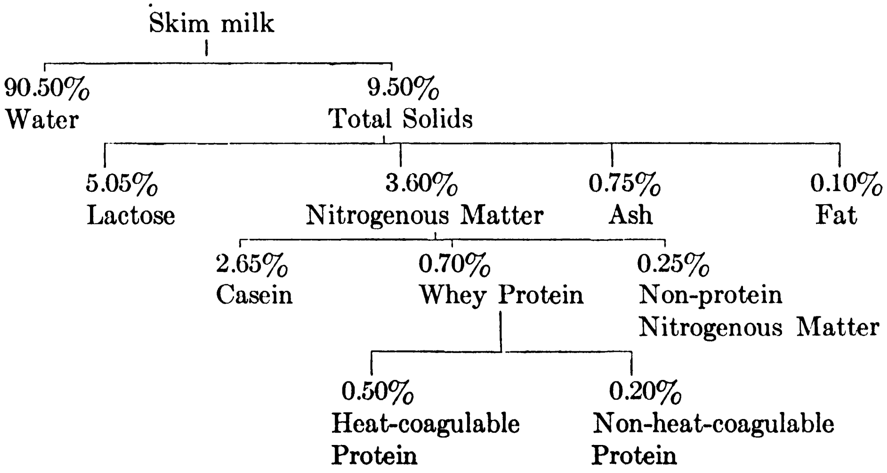
## SKIM MILK

Skim milk and cream are the products of passing whole milk through a cream separator. Since this process is conducted for the purpose of concentrating the milk fat, which is the component of greatest sales value, an effort is made to remove all fat possible from the skim milk portion. The fat of skim milk from the separator is usually less than 0.01 percent by the Babcock test. However, since dried skim milk (approximately a 1 to 10 concentration of skim milk) usually contains about 1.00 percent extractable fat, it is evident that the Babcock test does not detect all the fat in skim milk, hence, it is a sound assumption that the fat content of fluid skim milk is of the order of nearly 0.10 percent. Skim milk produced by hand-skimming is rarely found as a commercial product, therefore, its somewhat greater fat content need not be considered here.

The total nitrogenous matter of skim milk, calculated as 6.38 times the total nitrogen, is usually between 3.50 and 4.10 percent, the precise value depending mostly on the breed of the cow that produced the milk. Approximately 75 percent of the nitrogenous matter is coagulable by acetic acid and is considered to be casein.[32] An additional 13 percent is coagulable by heat. Trichloroacetic acid precipitates about 93.7 percent of the total nitrogenous matter of skim milk, and hence, about 5.7 percent of the total nitrogen represents protein not coagulable either by heat or by acetic acid. The remaining 6.3 percent is considered to consist of nitrogenous compounds of non-protein nature.

The lactose content of sweet skim milk is usually approximately 5.0 percent. If part of the lactose has been converted to lactic acid by souring of the milk, the sum of the lactose and lactic acid will be equal to the lactose present before souring, since there is no loss nor gain in the conversion. The ash content of skim milk is 0.72 to 0.80 percent: the ash does not include all of the salts of milk, since the citrates and some other salts are volatile at the temperature of ashing.

The composition of a typical separator-skimmed milk is as follows:

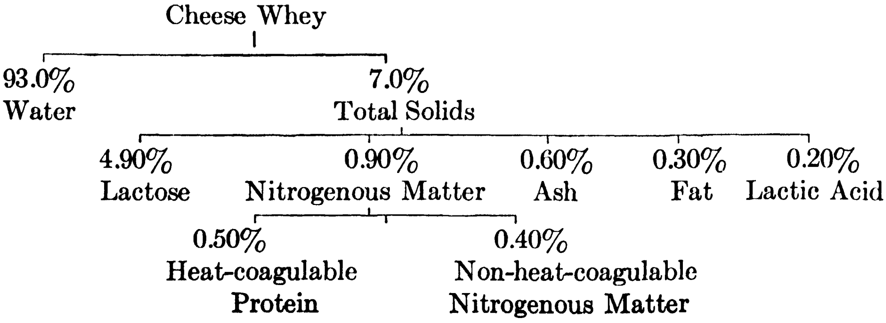


## WHEY

Whey is the greenish, yellow fluid remaining after the curd has been removed from either whole or skim milk. The casein may have been coagulated by rennet, in which instance the main product is cheese or rennet casein; or it may have been coagulated in skim milk by means of acid, either added or produced in the milk by fermentation of the lactose, in which instance the main product is commercial casein or cheese of the cottage type. Often, in making cottage cheese, both rennet and acid are used. When cheese is made from whole milk, the whey may contain as much as 0.70 percent fat. It is the usual practice to run such whey through a separator to remove as much of the fat as possible, as whey cream. This cream is usually added to cream destined for making into butter, but, since whey cream differs from ordinary cream in its whipping properties and in its response to sterilization treatments, it has somewhat unique value as a source of fat in certain processed foods (discussed in Chapter 9). Both separated whey and whey derived from skim milk contain approximately 0.10 percent fat.

Differences found in the compositions of wheys are attributable mainly to the method used in the coagulation of the casein, and to the extent to which fermentation of the lactose has been allowed to progress. When the coagulating agent is rennet, the calcium and phosphorus of the casein complex remain for the most part with the curd. The ash content of the whey is therefore less than it is when the coagulating agent is acid, which transfers part of the phosphorus and most of the calcium to the whey. Some fermentation is a requisite part of most processes of cheese making, the main product being lactic acid, which is formed gram-per-gram from the lactose. Lactic acid is formed in greater quantity in the making of "self-sour" casein. The formation of gases and volatile acids by fermentation reduces the lactose percentage and, in such instances, the increase in acidity, calculated as lactic acid, is not equivalent to the decrease in lactose.

The compositions of typical wheys are as follows:



Casein Whey

- 93.0% Water
- 7.0% Total Solids
  - 5.10% Lactose
  - 1.00% Nitrogenous Matter
    - 0.60% Heat-coagulable Protein
    - 0.40% Non-heat-coagulable Nitrogenous Matter
  - 0.70% Ash
  - 0.10% Fat

## BUTTERMILK

The word *buttermilk,* as earlier used, denoted the highly acid fluid byproduct of the churning of sour cream to make butter. Later, when it became more and more the custom to make butter from sweet cream, two varieties of buttermilk were recognized—sour-cream buttermilk and sweet-cream buttermilk. Then, too, the name buttermilk was applied to the beverage made by the lactic fermentation of skim milk. In this book, the simple term buttermilk, differentiated frequently as sweet-cream and sour-cream buttermilk, is applied only to the byproduct of making butter; the fermented beverages prepared from skim milk are designated specifically as cultured buttermilk, Bulgarian buttermilk, acidophilus milk, etc., and are discussed later in Chapter 2.

Sweet-cream buttermilk is similar to sweet skim milk in gross composition, differing in being slightly more dilute, and in containing slightly more fat. Since the proportion of water is slightly greater, the proportions of the component solids, other than fat, are correspondingly less, this being apparent chiefly in the percentages of nitrogenous matter and lactose. The nitrogenous matter of buttermilk differs in its characteristics from that of skim milk, as will be pointed out in the following paragraphs. The composition of a typical sour-cream buttermilk differs from that of the typical sweet-cream buttermilk given below, principally in that it contains 0.5 percent lactic acid and correspondingly less lactose.

Sweet-Cream Buttermilk

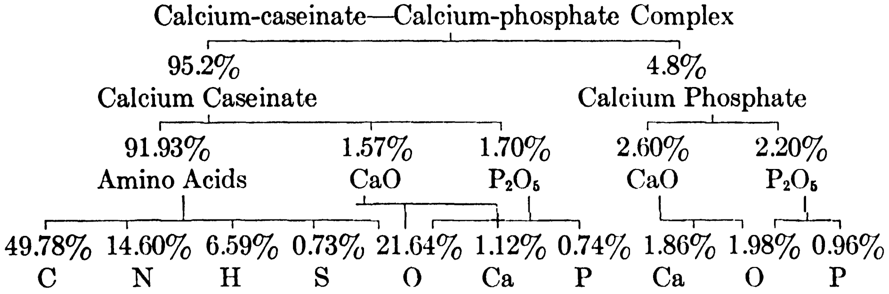
- 91.00% Water
- 9.00% Total Solids
  - 4.50% Lactose
  - 3.40% Nitrogenous Matter
  - 0.70% Ash
  - 0.40% Fat

## CASEIN

Casein, the principal nitrogen-containing compound in milk, exists in milk as a calcium compound with which is associated a small proportion of a calcium phosphate, which may be dicalcium phosphate, tricalcium phosphate, or both. The British term "caseinogen" refers to the casein complex as it occurs in milk. The calcium and phosphorus of the calcium phosphate are called "inorganic calcium" and "inorganic phosphorus" to distinguish them from the "organic calcium" and "organic phosphorus" of the calcium caseinate portion of the complex.

Casein coagulated by means of rennet has the same gross and ultimate composition as the casein complex of milk, but certain of its characteristics are different, as will be shown later. Rennet-coagulated casein is usually called rennet casein or paracasein. Casein, precipitated at pH 4.1 to 4.6 by acid, has been freed of the associated calcium phosphate and of the calcium combined directly with the protein. Hydrogen has displaced the calcium and, hence, acid-precipitated casein may properly be considered an acid, though it is a very weak acid. It is usually called casein, but is sometimes designated free casein, acid casein or acid-precipitated casein, to distinguish it from the more complex parent substance existing in milk.

The ultimate composition of the casein complex of milk is shown in the diagram below.



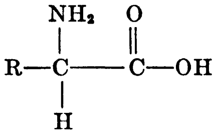
Although casein from any source appears to have the same ultimate composition, casein has been fractionated by means of acidified ethanol into portions that differ one from another in ultimate composition and in other characteristics.[27] The fractions recombined in the proper proportions gave a substance indistinguishable from the unfractionated casein, however. This indicates that components differing in chemical composition had been separated by merely physical means. Fractions differing in percentage of phosphorus have been separated by use of ammonium chloride solution.[10, 11] Centrifugal fractionation produces fractions that are, for the most part, of apparently the same elementary composition and specific gravity,[16] but there appears to be a small proportion of the protein that has a composition differing slightly from the main body. The slight differences in proportions of amino acids in caseins from cow milk and human milk[46] are evidence that not all caseins are of identical composition. The above facts are difficult to reconcile, but it seems at least probable that molecules of casein differ slightly in the proportion of amino acids, in the type of linkages between amino acids, and in relative arrangement of amino acids, but that in elementary composition and for most practical purposes casein may be considered homogeneous.

Centrifugation of milk about 24 hours old has shown that the colloidal protein present may exist as particles of from 2 to 12 different and definite sizes.[16] Usually, about 6 sizes are found. These dispersed particles are spherical in shape and consist of 1 volume of water-free protein and 3 volumes of solvate liquid. They appear to be multiples of a unit particle having a diameter of approximately 66 millimicrons and an apparent molecular weight of approximately 33 million. The predominant particle has an apparent molecular weight of 530 million; the largest particle observed, 15 billion.

Most of this protein is chemically homogeneous, but there is evidence that there is a small portion that differs slightly in elemental composition from the main body. The colloidal protein appears to be a chemical combination of casein, calcium and the phosphate radical in definite proportions.

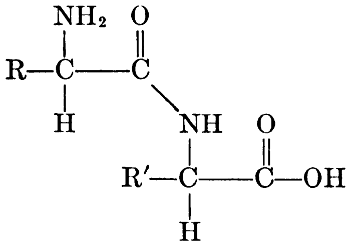
The distribution of sizes of the colloidal protein is inconstant and is affected by such factors as aging of the milk, heating, and the addition of surface-active materials. The precipitation of casein by means of acid and its redispersion results in particles greatly reduced in size. They are in the weight ratios of 1:2:4, the greatest portion having the smallest apparent molecular weight of between 75,000 and 100,000, provided the casein has not been heated above body temperature during the process of dispersing it in a buffer solution.[41] Dispersed at 105°F or above, it associates or polymerizes to particles of approximately 188,000 apparent molecular weight. Casein treated with hot, acidified ethanol has an apparent molecular weight of approximately 375,000. These three substances of different particle size are distinguishable one from another by serological reactions.[6] It has been calculated from analytical data that the probable formula weight of the smallest of these particles is 98,000.[7] This value is approximately three times the molecular weight of what appears to be the smallest casein unit, since cryoscopic measurements on solutions of casein in phenol[3] and in aqueous solutions of urea[5] have indicated values of 25,000 and 33,600, respectively, and since ultracentrifugal methods have shown that acid-precipitated casein dispersed in dilute buffer solutions has an apparent molecular weight of 32,000.[8] It should be clearly understood that redispersed acid-precipitated casein is, both chemically and physically a different substance from the complex colloidal protein of milk.

Free casein consists of alpha-amino acids of the general formula



$$R-\underset{\underset{H}{|}}{\overset{\overset{NH_2}{|}}{C}}-\overset{\overset{O}{\|}}{C}-OH$$

combined, for the most part, through peptide linkages thus:



Phosphoric acid is the linking group between some of the hydroxyamino acids. The calcium of calcium caseinate is joined through carboxyl or phosphoric groups. It should be noted that casein may combine with strong alkalies through these same groups, or with acids by addition to amine groups. In both cases, the compound formed is soluble in water.

TABLE 2 *Amino Acids in Casein*

| Common Name | Chemical Name | % by Weight | Molal. % (calc.) |
|---|---|---|---|
| Arginine | α-amino-δ-guanidovaleric acid | 3.84 | 2.56 |
| Histidine | α-amino-4-imidazolepropionic acid | 2.57 | 1.92 |
| Lysine | α,ε-diaminocaproic acid | 7.66 | 6.07 |
| Leucine | α-aminoisocaproic acid | 10.1 | 8.9 |
| Isoleucine | α-amino-β-methylvaleric acid | 7.0 | 6.2 |
| Cystine | β,β′-dithiodialanine | 0.34 | 0.16 |
| Methionine | α-amino-γ-methylmercaptobutyric acid | 2.93 | 2.27 |
| Tyrosine | α-amino-β-parahydroxyphenylpropionic acid | 6.32 | 4.05 |
| Tryptophane | α-amino-3-indolpropionic acid | 1.26 | 0.72 |
| Valine | α-aminoisovaleric acid | 6.8 | 6.7 |
| Phenylalanine | α-amino-β-phenylpropionic acid | 5.52 | 3.87 |
| Alanine | α-aminopropionic acid | 3.7 | 4.8 |
| Serine | α-amino-β-hydroxypropionic acid | 6.14 | 6.77 |
| Threonine | α-amino-β-hydroxybutyric acid | 4.45 | 4.33 |
| Glutamic acid | α-aminoglutaric acid | 21.8 | 17.2 |
| Aspartic acid | α-aminosuccinic acid | 6.8 | 5.9 |
| Glycine | α-aminoacetic acid | 0.5 | 0.8 |
| Proline | 2-pyrrolidinecarboxylic acid | 8.7 | 8.8 |

Eighteen different amino acids have been definitely identified as components of casein and their proportions determined. Caseins from milks of different animals contain approximately the same proportions of amino acids, but it is probable that the amino acids are somewhat differently arranged in casein molecules from milks of different species. Sulfur is present in two of the amino acids, cystine and methionine.

Determinations of the amino acids in casein have been made by many investigators and by many methods. Since these determinations have been made on caseins that were purified and hydrolyzed, both procedures usually causing some decomposition, the values found are, for the most part, in poor agreement. The percentages given in Table 2 have been selected as the most probable values.

Through hydrolytic action induced by enzymes, by acids, or by alkalies, casein may be split into a great variety of less complex combinations of amino acids, and ultimately into the amino acids themselves. Especially in the alkaline hydrolysis, the phosphoric groups are freed and the amino acids lose sulfur and nitrogen. Some of the amino acids set free are of essential nutritional value and one—glutamic acid—has an important use as an intensifier of flavor in food products. The preparation of hydrolyzates for food use should be made with either enzymes or acid; the use of alkali results in the formation of undesirable products.

## THE NITROGENOUS SUBSTANCES OF WHEY

The heat-coagulable protein of whey has been considered to consist of albumin and globulin, the albumin being the portion insoluble in saturated ammonium sulfate solution and soluble in neutral, saturated magnesium sulfate solution, the globulin being the portion insoluble in half-saturated ammonium sulfate solution and insoluble in saturated magnesium sulfate solution. These protein fractions have been further fractionated into substances that differ among themselves in composition and physical properties. The results of this secondary fractionation have been verified and summarized by Sørensen and Sørensen.[39] From the *lactoglobulin fraction* have been obtained: (a) a green fraction, or *lactomucin*, which is a sticky, green substance with low solubility in water and neutral salt solutions; and (b) a red fraction which is freely soluble in water and half-saturated ammonium sulfate solution. From the *lactalbumin fraction* have been obtained: (c) Palmer's crystalline globulin-like substance[29] which has a very low solubility in water and high solubility in weak salt solutions, and at pH 6 to 7 can be precipitated as crystals by ammonium sulfate; (d) a gelatinous substance which is easily soluble in water, but insoluble in half-saturated ammonium sulfate solution at pH 4.6, and which can be precipitated at pH 6 to 7 as crystals by ammonium sulfate; and (e) an "insoluble substance."

Substances (c) and (d) are probably of almost identical composition and are interconvertible; substance (c) is not sharply separable from (d).

The *lactalbumin fraction* differs from free casein in elementary composition in that it contains nearly three times as much sulfur and only a trace of phosphorus; the *lactoglobulin fraction* contains a slightly greater proportion of sulfur than does casein and about one-third as much phosphorus. The three proteins differ also slightly in the proportions of the component amino acids. It should be noted that the micellar weight of the whey proteins after isolation is of the order of 12,000 to 25,000, but that in milk the micellar weight is probably less than 1000.[36] The differences among the three proteins of milk are not as sharp as could be desired. There is the possibility that those of smaller apparent molecular size are the precursors of the larger ones, phosphoric acid and calcium phosphate acting to some extent as connecting links between units.

Whey may be heated for a short time at 149°F without hydrolysis or denaturation of its protein, but boiling of whey for 5 minutes causes complete denaturation. The proportion of the denaturated protein that coagulates depends on the pH value of the whey, the maximum quantity of protein coagulating in the range of pH 4.75 to 4.80.[32] For the purposes of this book, it is not necessary to know whether the heat-coagulable nitrogenous matter of whey is chiefly albumin or chiefly globulin and it will be considered in subsequent chapters merely as whey protein.

Trichloroacetic acid and tungstic acid precipitate from whey still other protein-like substances, which are probably peptone or proteose in nature and either were not built into true proteins during the formation of milk or were split off from proteins in the process of their isolation. There are in whey other nitrogenous substances that are not precipitable by any of the usual means or reagents; some of these have been identified, as creatin, creatinin, urea, uric acid, amino acids and ammonia.

## BUTTERMILK PROTEIN

Although buttermilk protein has the same elementary composition as does skim milk protein, it differs to such an extent in its physicochemical properties that it requires separate consideration. In the churning of cream to produce butter, the fat globules of the cream are caused to coalesce to form a continuous phase of fat in which aqueous serum is present as droplets. In this process the membrane, which enveloped and isolated each fat globule and is partly globulin-like and partly phospholipid in nature, is ruptured and dispersed in the buttermilk.[30] This membrane protein is unlike any other milk protein in physical properties. It differs from them in amino acid structure and in serological tests, and contains no phosphorus.[31, 44, 45] Furthermore, the other milk proteins have undergone degradation, presumably through the incompletely understood adsorptive and desorptive action that took place during the churning. Addition of acid to buttermilk causes the precipitation of a fine, soft curd that is very difficult to filter. On drying, this curd darkens in color, probably because of fat or phospholipids associated with the protein.

## LACTOSE

Lactose is the most important of the solids of dairy byproducts from the standpoint of content. The quantity present in sweet skim milk, buttermilk and whey is approximately 5 percent. In soured milks and wheys, the lactose percentage is decreased, the decrease being accounted for, for the most part, by lactic acid formed in the fermentation and to a small extent by volatile acids, such as acetic and butyric acids, and by carbon dioxide.

Lactose is a mildly sweet, relatively slightly soluble disaccharide, which on hydrolysis becomes a much sweeter and much more soluble mixture of equal proportions of glucose and galactose. Lactose is a reducing sugar and on mild oxidation, as by bromine, is converted into lactobionic acid. This acid on hydrolysis yields galactose and gluconic acid; hence, the oxidation must have taken place on the glucose portion of the lactose molecule and lactose must be a galactosyl glucose, the union of the two monosaccharides involving the aldehyde, or reducing, group of galactose. If oxidation and hydrolysis are carried on together, the products are gluconic and galactonic acids.

The formula of lactose is usually written $C_{12}H_{22}O_{11} \cdot H_2O$, since this is the composition of the common, crystalline lactose of commerce. This form is more specifically designated as alpha-lactose hydrate, an alpha-lactose anhydride being easily made by dehydrating the hydrate at a temperature between 149° and 200.3°F, preferably under reduced pressure, and a form known as beta-lactose anhydride being produced by crystallization of lactose from aqueous solutions at temperatures above 200.3°F. The alpha and beta forms are believed to differ slightly in structure, but the more striking differences are in solubility and in the effect of their solutions on polarized light passing through them. Beta lactose is much more soluble in water than is alpha lactose, as shown in Table 3; the specific optical rotation of beta lactose is +35.0°, that of alpha-lactose +89.4°.

The forms of lactose present in water solution, the forms crystallizing from solution at different temperatures, and the rates of crystallization[18, 24] at different temperatures are of technical interest; yet the statements on these points in books on dairy products are often misleading. Hence, the pertinent facts and their explanation are discussed here in detail.

When alpha-lactose hydrate is dissolved in water at ordinary temperatures, its specific rotation changes gradually from an initial value of +89.4°

to a final, constant value of +55.5°. When beta-lactose anhydride is dissolved similarly, its specific rotation changes gradually from an initial value of +35.0° to a final constant value of +55.5°. At temperatures above 158.0°F the approach to the equilibrium value is so rapid as to appear to be instantaneous and the value differs very slightly from +55.5°F. Calculations based on these rotations show that, in an aqueous solution of lactose in equilibrium at ordinary temperatures, 62.25 percent of the lactose is in the beta form and 37.75 percent is in the alpha form. This ratio differs slightly with differences in temperature, but all water solutions of lactose in equilibrium at any temperature contain the two forms in approximately the ratio of 62.25/37.75, or 1.65/1.00. If these solutions are deprived of water practically instantaneously, as may be done by means of a spray or drum dryer, the dry product is a mixture of the alpha and beta forms in the equilibrium ratio.

TABLE 3. *Solubilities of Lactose*

| Temperature, in °F and °C | | Initial Solubility | | Final Solubility, g. per 100 g. water |
|---|---|---|---|---|
| | | Alpha, g. per 100 g. water | Beta, g. per 100 g. water | |
| 32.0 | 0 | 5.0 | 45.1 | 11.9 |
| 59.0 | 15.0 | 7.1 | | 16.9 |
| 77.0 | 25.0 | 8.6 | | 21.6 |
| 102.0 | 39.0 | 12.6 | | 31.5 |
| 120.2 | 49.0 | 17.8 | | 42.4 |
| 138.4 | 59.1 | | | 59.1 |
| 146.8 | 63.9 | | | 64.2 |
| 147.2 | 64.0 | 26.2 | | 65.8 |
| 164.3 | 73.5 | | | 84.5 |
| 165.2 | 74.0 | 34.4 | | 86.2 |
| 174.4 | 79.1 | | | 98.4 |
| 189.0 | 87.2 | | | 122.5 |
| 190.8 | 88.2 | | | 127.3 |
| 192.2 | 89.0 | 55.7 | | 139.2 |
| 212.0 | 100.0 | | 94.7 | 157.6 |
| 224.6 | 107.0 | | | 177.0 |
| 250.7 | 121.5 | | | 227.0 |
| 272.5 | 133.6 | | | 273.0 |
| 281.4 | 138.8 | | | 306.0 |

Solutions saturated with respect to lactose at 200.3°F, prepared either by evaporating dilute solutions or by shaking with crystalline lactose until equilibrium is attained, are saturated both with respect to the alpha and to beta forms. This does not mean that the solubilities of the two forms are the same at this temperature, but that the ratio of the solubilities at this temperature is the same as the equilibrium ratio of the two forms in solution.

If the solution is concentrated further at 200.3°F, it becomes supersaturated with both forms and seeding with a few crystals of either form will cause that form to crystallize. Since the equilibrium between the forms in solution is thus disturbed, a sufficient portion of the other form will change to the form crystallizing to reestablish equilibrium. Crystallization and reestablishment of the equilibrium ratio will continue until the solution is no longer supersaturated.

If, however, a solution is saturated at a temperature higher than 200.3°F, it is saturated with respect to the beta form, but not saturated with respect to alpha lactose. Further concentration produces a solution supersaturated with respect to beta but still not saturated with respect to alpha. From such a solution only beta-lactose anhydride will crystallize, equilibrium in the solution being reestablished by change of alpha to beta. Alpha lactose added to such a solution will dissolve and an equivalent quantity of beta-anhydride will crystallize, a phenomenon on which is based a patented process for the production of beta lactose (described in Chapter 11). It is true that solutions of lactose above 200.3°F may be sufficiently highly concentrated to become supersaturated with respect to both alpha and beta and that seeding with alpha-lactose crystals will cause alpha-lactose hydrate to crystallize from such solutions, but subsequent addition of beta lactose will cause beta to crystallize and the crystallized alpha to redissolve and recrystallize in the beta form.

The situation is reversed for solutions of lactose below 200.3°F, a saturated solution being saturated with alpha, but not with beta. A slightly supersaturated solution is supersaturated only with respect to alpha, the form crystallizing is alpha-lactose hydrate, equilibrium in solution being reestablished by the change of beta to alpha.

Since the rate of reestablishment of alpha-beta equilibrium in solution is practically instantaneous above 158.0°F, the quantity of lactose crystallizing above that temperature in a definite time is a function only of the degree of supersaturation at the temperature of the solution. At lower temperatures, especially near 32.0°F, the rate of change of beta to alpha lactose in solution is much slower than the normal rate of crystallization and becomes a determining factor in the quantity of sugar crystallizing in a definite time. The over-all rate of crystallization for the first two and one-half hours after cooling a solution of lactose that has been saturated at boiling temperature is greater at 86.0°F than at lower temperatures.[43] The most efficient method of crystallizing alpha-lactose hydrate is to cool the solution to 86.0°F, to seed it and stir at 86.0°F for about three hours, then to cool to about 68.0°F, and to hold for at least three or four hours before filtering out the sugar.

Crystalline alpha-lactose hydrate is stable in air at ordinary temperatures; between 149.0 and 200.3°F in dry air it slowly loses water, changing to alpha-lactose anhydride. Above 200.3°F, the hydrate loses water to become the beta anhydride. Both anhydride forms are stable in dry air at temperatures either above or below 200.3°F, but in moist air below 200.3°F both forms change to the alpha hydrate, and above 200.3°F in moist air the alpha anhydride changes to the beta anhydride.

Solubilities of lactose in water are given in Table 3.[18, 24] It should be clearly understood that, though initially beta lactose is much more soluble than alpha lactose, the final, or equilibrium, solubility is the same and the composition of the equilibrated solution the same, whatever the form initially dissolved. Statements not in agreement with these facts should be disregarded. Because of the slow and limited solubility of alpha lactose and the hardness and sharp edges of its crystals, lactose present in dairy products is frequently the cause of a condition known as "sandiness."

## ASH CONSTITUENTS

The residue remaining after heating milk to a temperature sufficiently high to volatilize the water and the carbon and nitrogen compounds present is known as the ash of milk and consists of salts, some previously existing as salts and some formed by combination of metallic and non-metallic ions during the evaporation of the water. Analytically, the ash of milk may be considered as consisting of combinations of metallic oxides with non-metallic oxides and metals with chlorine. Since in an analysis all the metals are given as oxides, the sum of the weights of the metallic oxides, the non-metallic oxides and chlorine will be greater than the weight of the ash by an amount equal to the oxygen equivalent of the chlorine. Normally, the metallic oxides are in excess and hence a solution of milk ash in water is alkaline. Calcium and phosphorus are considered the most important elements in milk ash, since they are essential in nutrition; these elements and potassium are present in milk in amounts large in proportion to the other metals and non-metals. The calcium and phosphorus of milk ash include the "organic" calcium and phosphorus of the casein complex, but, since this is retained by curd coagulated by rennet, it is not included in the ash of whey from rennet cheese.

Ash constituents are found in the following percentages in typical skim milk or buttermilk:—

| $K_2O$ | $CaO$ | $Na_2O$ | $MgO$ | $Fe_2O_3$ | $P_2O_5$ | $Cl$ | $SO_3$ |
|---|---|---|---|---|---|---|---|
| 0.188% | 0.151% | 0.075% | 0.018% | 0.001% | 0.182% | 0.107% | 0.029% |

In cheese whey from rennet cheese, the percentages of these components are practically the same as given above, except that $CaO$ makes up only 0.071 percent and $P_2O_5$, 0.110 percent. The ash of casein whey and whey from sour milk cheese is like that of skim milk, except that, when hydro-

Table 4. *Water-soluble Vitamins in Milk and Milk Byproducts*
$\gamma$/g.

| | Thiamine | Riboflavin | Ascorbic Acid | Nicotinic Acid | Pantothenic Acid | Biotin | Pyridoxine | Inositol | Folic Acid | Choline |
|---|---|---|---|---|---|---|---|---|---|---|
| *Whole Milk* | | | | | | | | | | |
| Normal, fresh | 0.405<br>0.41–0.48 | 2.00<br>1.77 | 21.8 | 0.91 | 3.1<br>2.9 | 0.047<br>0.050 | 0.67 | 180 | <0.05 | 149 |
| References | (19, 25) | (20, 26, 14) | (17, 23, 42) | (22) | (22, 9) | (22, 9) | (21) | (9) | (9) | (22) |
| Evaporated | 0.58 | 3.57 | 4.0–27.6 | 1.96 | 6.2 | 0.090 | | | | 262 |
| References | (34) | (14) | (47) | (22) | (22) | (22) | | | | (22) |
| Dried | 2.9 3.15 | 15.5 15.44 | 93 97.1 | 6.9 6.7 | 24 | 0.41 | 3.3 | 1400 | <0.4 | 1093 570–1000 |
| References | (4, 35) | (14, 35) | (35, 47) | (22, 35) | (9, 35) | (9, 35) | (35) | (9) | (9) | (22, 35) |
| *Skim Milk* | | | | | | | | | | |
| Normal | 0.42 | 1.58 | | | | | | | | |
| References | (4) | (14) | | | | | | | | |
| Dried | 3.73 | 19.8 | 62.7 | 8.96 | 37.1 | 0.350 | | | | 801 |
| References | (4, 12, 28) | (1, 13, 14, 15, 28, 37, 40) | | (22) | (22) | (22) | | | | (22) |
| *Whey* | | | | | | | | | | |
| Normal | | 1.24 | | | | | | | | |
| References | | (14) | | | | | | | | |
| Dried | 1.64 | 22.6 | | | | | | | | |
| References | (12) | (13, 14, 15, 38, 40) | | | | | | | | |
| *Buttermilk* | | | | | | | | | | |
| Cultured | | 1.56 | | | | | | | | |
| References | | (14) | | | | | | | | |
| Natural, dried | | 33.0 | | | | | | | | |
| References | | (13, 14, 15, 37) | | | | | | | | |

chloric or sulfuric acids are used as precipitants of casein, the Cl or $SO_3$ are increased and the percentages of the other ash constituents are slightly decreased by the dilution with the acid added.

The percentages of ash constituents of concentrated milk products may be calculated by multiplying the above values by the concentration factor. In rennet cheese, however, the ash is concentrated from 5- to 10-fold, the degree depending on the method of manufacture. In making dried whey the ash is concentrated 9- to 10-fold; if part of the lactose is removed before drying, as in making whey protein powder, the concentration of ash may be as great as 22-fold. If an acid whey, such as grain-curd-casein whey, is neutralized with NaOH or $Ca(OH)_2$, there is formed a precipitate that consists of $Ca_3(PO_4)_2$ mixed with some whey protein.[2] A product of this sort has been added to dried whey to increase the calcium and phosphorus percentages, thus increasing the nutritive value of the dried whey for therapeutic use.

## VITAMINS

The vitamins known to be present in milk include vitamins A, D, and E, thiamine, riboflavin, ascorbic acid, nicotinic acid, pantothenic acid, biotin, pyridoxine, inositol, folic acid, $B_{12}$, and cho ine. Since vitamins A, D, and E are associated with the milk fat, which is a very minor constituent of most dairy byproducts, these vitamins are not considered here.

Some typical values for the content of water-soluble vitamins in whole milk, milk byproducts, and their concentrated forms are listed in Table 4. In instances in which several references are given, the values are averages of those found by several investigators. Values for whole milk and concentrated whole milks are included as a basis for comparison in order that the effects of processing and other influences may be apparent. Milk is rated as a good source of only riboflavin and biotin, in comparison with other sources of water-soluble vitamins.[9]

Thiamine is destroyed to some extent in processing and in storage. Riboflavin is destroyed by exposure to light, as much as 50 percent being lost from a bottle of milk exposed to full sunlight for 4 hours. The destruction of ascorbic acid by oxidative processes is continuous, the rate of destruction being increased by the catalytic action of salts of copper and iron and by fermentation. The other vitamins appear to be relatively stable under conditions to which milk is exposed during processing and storage.[22]

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# 2

# BEVERAGES FROM BYPRODUCTS

## INTRODUCTION

The principal beverages made commercially from dairy byproducts are cultured buttermilk and chocolate-flavored milk, both prepared from skim milk, but frequently containing added fat. Bulgarian cultured milk, acidophilus milk, kefir, and yoghurt are made in small quantities in some sections of the United States, but utilize only an insignificant quantity of skim milk. A sterilized caramel-flavored milk was made for the use of the armed forces during World War II; this however, has not been generally available and is no longer being made. Beverages based on whey have been made only on an experimental scale. Caramel milk and several whey beverages are discussed in Chapter 8.

## CULTURED BUTTERMILK[3, 4, 9]

The unqualified term, buttermilk, denotes the fluid byproduct of the churning of cream into butter, and it is usually understood to refer to buttermilk obtained in the churning of sour cream. In the churning of sweet cream, there is produced a buttermilk known generally as sweet cream buttermilk. Cultured buttermilk resembles the buttermilk derived from sour cream, but it is not, strictly speaking, buttermilk, since it is made by fermentation of skim milk, partially skimmed milk, or, less commonly of sweet cream buttermilk, reconstituted soluble dried skim milk,[8] or reconstituted plain condensed skim milk. The fermentation process used is, however, similar to that used in ripening cream for churning into butter, and cultured buttermilk may be churned in order that flakes or granules of butter may be present, or lightly churned cream added for the same purpose.

**Cultures.** The cultures used in making cultured buttermilk are usually the same as those used as starters in making Cheddar cheese and in ripening cream for buttermaking. They are mixed cultures, containing *Streptococcus lactis* as the principal species of bacteria, and producing lactic acid and small quantities of compounds that give a pleasing flavor and aroma to the product.

It is possible to prepare a culture suitable for use from a clean-flavored sour milk, but it is much preferable to purchase a culture from a commercial laboratory. The culture should be propagated in sterile milk in duplicate, one culture being held in reserve as a stock culture, the other used in the preparation of the starter or bulk culture to be used for inoculation of the batches of milk. In spite of precautions, the bulk culture may become contaminated with organisms that produce gas or undesirable flavors or the flavor-producing bacteria may become inactive. The maintenance of a stock culture makes the overfrequent purchase of new cultures unnecessary.

**Propagation of Stock Cultures.** Fresh, good-quality skim milk is added to small flasks or heat-resistant glass bottles to one-third or one-half their capacity, the containers plugged with non-absorbent cotton, and the milk sterilized by heating in an autoclave or steam-pressure sterilizer for 15 minutes at 15 pounds pressure. The milk is cooled to approximately 70°F, and the culture added by means of a sterile pipet in the proportion of 1 to 3 percent. The inoculated milk is incubated at about 70°F for 18 to 24 hours. It should be in a curdled condition at the end of the incubation period. New transfers are made similarly each day. After several transfers of a newly obtained culture, it should be in an active condition suitable for inoculating bulk culture. The portions of the incubated cultures remaining after transfer should be examined for flavor and aroma, and any deteriorated culture and the transfer from it should be discarded.

**Propagation of Bulk Culture.** Bulk culture should be prepared in quantity sufficient to furnish inoculum equivalent to from 1 to 5 percent of the volume of milk to be fermented. As much as 10 percent may be used for a rapid fermentation, or as little as 0.1 percent if the culture is especially vigorous or a slow fermentation is desired.

Stainless-steel cans of 2- or 5-gallon capacity made especially for preparing bulk culture may be purchased from dairy supply firms. Cabinets for heating milk in these cans, and incubators of suitable dimensions for holding several cans at one time, may also be purchased. Covered enamelware or well-tinned cans may be substituted for stainless-steel cans.

The fresh skim milk to be used in making bulk culture should be placed in the starter cans and heated at 185 to 212°F in boiling water or steam for an hour. The heated milk is cooled to about 70°F and 0.2 to 0.5 percent of active culture added and thoroughly mixed with the milk. The inoculated milk is incubated at 70°F for 14 to 16 hours. The proportion of inoculum used should be sufficient to cause the development of 0.75 to 0.85 percent of acid by the end of the inoculation period. The bulk culture is then ready for use in inoculating a batch of milk. If it should not be possible to use the culture immediately, it should be refrigerated until used.

**Preparation of Cultured Buttermilk.** The manufacture of uniformly high quality cultured buttermilk requires cultures maintained in active and uncontaminated condition, a supply of uniformly fresh, clean milk, equipment that can be thoroughly cleaned and sterilized, and a standardized manufacturing procedure.

Fresh skim milk is the preferred starting material. Reconstituted plain condensed or dried skim milk of good quality may be used, but the poorer grades give a cultured buttermilk having a cooked flavor. If milk containing appreciable quantities of fat is used, the fat rises during the fermentation and becomes so viscous when the fermented milk is cooled that difficulty is experienced in redistributing it throughout the finished product. If it is desired to enrich the milk in fat, it is preferable to do it after fermentation by mixing cream with the milk while the curd is being broken up. Consumer preference studies have shown no advantage in the addition of fat.[8] Sweet cream buttermilk can be used successfully if it has no objectionable flavors, and if the air incorporated during the churning process is expelled in the heating that precedes inoculation.

Vats specially constructed for making cultured buttermilk may be purchased. These are of glass-lined or stainless steel construction and are essentially well-insulated pasteurizers. Insulated vats equipped with covers, agitators, and either coil or jacket heaters may be used. Tinned vats are undesirable because of the rapid destruction of the tin coatings by the lactic acid of the cultured milk.

The milk is heated to 180 to 190°F for 30 minutes with frequent stirring and cooled to 68 to 72°F. One to five percent of bulk culture is then added, the milk thoroughly stirred, the vat covered, and the milk incubated at 68 to 72°F until it is curdled and the acidity is between 0.8 and 0.9 percent. Curdling before the acidity is in this range indicates the need of a larger proportion of starter. At the end of incubation it should be possible to separate the curd cleanly from the side of the vat and there should be little or no whey on the surface of the curd. The time required for incubation is usually between 14 and 24 hours. The curdled milk is cooled to 50°F or below, and, when cooled, stirred to break up the curd and give a smooth consistency to the product. The cultured milk is then bottled in ordinary milk bottles and kept below 50°F until distributed to customers.

The flavor of cultured buttermilk may be intensified by adding 0.15 percent of citric acid to the milk at the time of inoculation. For each 10 gallons of milk, 2 ounces of citric acid are dissolved in 1/2-pint of water. The solution is boiled for a few minutes to sterilize it, cooled, and added very slowly to the milk while it is being stirred, prior to incubation. Too rapid addition will cause local coagulation of the milk.

A churned buttermilk may be made by fermenting milk containing 1 or

2 percent fat according to the directions given above, cooling the curdled milk to 56°F, and then churning it until butter granules of the size of rice grains appear. Butter color may be added to increase the color of the butter granules, if local regulations permit. Another method is to prepare the cultured buttermilk from skim milk and to stir in butter granules while breaking up the curd. For each 10 gallons of buttermilk, 1 gallon of pasteurized 20 percent cream, or its equivalent in cream containing a greater percentage of fat, is ripened to contain 0.35 percent acid, butter color is added, and the cream cooled and churned until butter granules, of the size desired, are obtained. The granules are removed, washed with cold water, suspended in a little cold water, and put in a refrigerator until the particles of fat are firm. The granules are then removed from the water and mixed with the cultured buttermilk. Still another method is to allow butter oil, at 120 to 130°F, to drip through needle size holes into cold buttermilk while the milk is being stirred. The same result may be obtained by spraying the butter oil into the cold buttermilk. The granules produced by this method are in the form of discs.

Cream may also be stirred into cold cultured buttermilk during the breaking-up process without churning. This has the effect of decreasing the viscosity of the buttermilk and of imparting a rich flavor. Another method of decreasing the viscosity is to add 20 to 25 percent of pasteurized, sweet whole milk during the breaking up of the curd. This addition also appreciably decreases the acidity of the cultured milk.

Wheying off of cultured buttermilk is a defect sometimes encountered. This may be caused by heating the raw milk at too low a temperature—$<$ 180°F—by insufficient cooling before breaking up the curd, by too violent agitation during the breaking up of the curd, or by too small a percentage of milk solids in the product. Methods of controlling temperature and agitation are obvious; if the fault is in the lack of adequate milk solids, 1 to 2 percent of dry milk, or 5 to 10 percent of plain condensed milk may be mixed with the milk prior to the heating.

Salt in the proportion of 1 teaspoonful to 10 gallons may be added to cultured buttermilk to accentuate its flavor.

## ACIDOPHILUS MILK[3, 7]

Acidophilus milk is consumed mainly for therapeutic reasons. It is claimed that the growth of *Lactobacillus acidophilus*, the organism present in acidophilus milk, under conditions existing in the intestinal tract will replace undesirable putrefactive fermentations with a beneficial lactic acid fermentation. Lactose and dextrin have been shown to aid in the establishment of *L. acidophilus* in the intestinal tract; hence, it is a common practice to add either of these substances—more often, lactose—to acidophilus milk.

There is no valid reason for adding a mixture of the two substances; actually, the addition of a mixture defeats the purpose in view.

**Propagation of Cultures.** A pure culture of *L. acidophilus* should be obtained from a reliable source and mother cultures propagated, preferably in duplicate, by methods of sterile technic. Skim milk that has been sterilized by heating for 15 minutes under 15 pounds pressure is brought to a temperature of 100°F and inoculated with 1 to 2 percent of the pure culture. It is then incubated at 100°F until the milk is curdled and then placed in a refrigerator. Transfers should be made every 2 or 4 days by this method. As long as the culture produces a smooth curd having the typical acidophilus flavor, it is probably uncontaminated.

The bulk culture or starter should be prepared by inoculating with mother culture the necessary quantities of sterile skim milk in large flasks, and should be handled by the technic used in preparing the small mother cultures. When only small quantities of acidophilus milk are to be prepared, the portion of mother culture remaining after transfers have been made may be sufficient for bulk inoculation.

**Preparation of Acidophilus Milk.** Freshly separated, sweet skim milk is placed in a glass-lined or stainless-steel jacketed vat provided with a loosely fitting cover. For small batches, stainless-steel starter cans may be used. The containers should be not over three-fourths filled. The addition to the milk of 1 percent of glucose or 5 percent of tomato juice is of advantage in hastening the fermentation, but is not essential. The containers are covered and the milk is heated to above 190°F for an hour. It is then cooled to 100°F and inoculated with 1 to 5 percent of culture. The inoculated milk is mixed thoroughly and incubated at 100°F until it is curdled and has the desired acidity, which ordinarily requires 18 to 24 hours. It is then cooled in a refrigerator to 50°F, and 5 to 15 percent lactose or dextrin added. After standing for 5 minutes, the curd is broken up and the milk stirred until smooth. It is then bottled and stored in a refrigerator until used.

## BULGARIAN CULTURED MILK[3, 7]

This variety of cultured milk, often called Bulgarian buttermilk, was formerly used for therapeutic reasons, but has been replaced largely for this purpose by acidophilus milk. However, it is consumed by some people in preference to ordinary cultured buttermilk because of its greater acidity. It lacks the flavor and aroma of cultured buttermilk, is much more viscous, and may even be gelatinous or ropy. For these reasons, Bulgarian cultured milk is usually mixed with cultured buttermilk to make a product having a mild flavor and aroma, a sharp acidity, and a thick consistency. A mixture of equal parts of the two cultured milks is recommended, but other proportions may be used according to taste. It is not practicable to carry out the two fermentations simultaneously in a batch of milk because of the different temperature requirements of the bacteria involved.

The method of making Bulgarian cultured milk is nearly the same as that for making acidophilus milk, the only differences being that a pure culture of a different lactobacillus is used, fermentation is considered complete when the acidity is approximately 1.5 percent, and no lactose or dextrin is added to the fermented milk.

## KEFIR[6, 13]

Kefir is a self-carbonated, fermented beverage, which may be whole, partially skimmed, or skim milk, containing usually about 1 percent lactic acid and from 0.5 to 1.0 percent alcohol. It is a popular food drink in the area about the Caucasus Mountains. In Russia, a product similar to kefir, except that it contains a greater percentage of alcohol, is made from mares' milk and is known as kumiss. Kefir grains, which in the dry condition resemble brownish or coral-colored seeds and are of the size of wheat grains or slightly larger, constitute the fermenting agent. They consist principally of substances derived from milk, bacteria that produce lactic acid, and lactose-fermenting yeasts that produce alcohol and carbon dioxide. The percentages of lactic acid and alcohol in kefir can be varied by varying times and temperatures of fermentation. The carbonation is obtained by carrying out the later stages of fermentation in tightly stoppered bottles.

**Activation and Propagation of Kefir Grains.** Kefir grains may be kept in dry condition for a year or more and may be purchased in this form. To bring them into active condition, they should be soaked for 2 to 3 hours in water at 85 to 90°F, and then, for the same time and at the same temperature, in water containing 1 percent of sodium bicarbonate. The grains that are swollen, elastic and translucent and that tend to rise to the surface should be selected, rinsed in water, and added to a bottle of sterilized skim milk. The inoculated milk should be incubated for 24 hours at 65 to 70°F, and the grains transferred daily to fresh sterilized skim milk until gas bubbles are produced in the milk. The grains are then ready for use.

To maintain the grains in active condition and at the same time increase the quantity, they should be transferred into fresh, sterilized skim milk every 2 or 3 days and the cultures incubated at 65 to 70°F. In transferring, the grains should be poured on a clean, metal sieve and rinsed with cold water before adding them to the fresh milk. A cupful of wet grains to a quart of milk is a suitable proportion to use. The grains used in making the kefir can be re-used indefinitely, provided they do not become contaminated, but they increase only slightly in making the kefir.

If kefir grains are to be kept for an extended period, they should be dried. They are removed from the milk, thoroughly washed in cold water, spread on cheesecloth, and dried in the sun, or in circulating air at about 86°F.

**Preparation of Kefir.** Pasteurized milk containing 2 to 3 percent fat, or pasteurized skim milk, is cooled to 57 to 60°F. About $\frac{1}{4}$-pound of moist, active kefir grains is added for each gallon of milk and the milk is held at 57 to 60°F for 8 to 10 hours, the container being protected from contamination by means of cheesecloth or a loosely-fitted cover. The milk is stirred several times during incubation. It is then strained through clean cheesecloth and transferred to tightly-stoppered or capped, heavy-walled, glass pop bottles. It is then incubated for 24 hours longer at 57 to 60°F with frequent shaking to break up the curd. The bottles are then placed on ice or in a refrigerator to check the fermentation. For subsequent batches, the kefir itself may be used as inoculum, but the grains usually give more uniform results.

If it is found that this procedure gives a kefir too low in acid and alcohol, the quantity of inoculum in subsequent batches may be increased and the period of incubation in the bottles may be extended. If a higher proportion of alcohol to acid is desired, the quantity of inoculum should be decreased and the temperature of incubation increased to between 63 and 68°F. Since the characteristics of kefir depend on the vigor of the kefir grains and a number of other factors besides the size of inoculation and time and temperature of incubation, it is not feasible to give precise directions that will produce a product of definite composition; with a little experience it should be easy to alter conditions to produce a kefir having the properties desired.

**Imitation Kefir.** If kefir grains cannot be obtained, it is still possible to prepare a fermented milk similar in composition and properties to kefir. Ordinary bread yeast may be used to produce alcohol in milk, provided cane sugar is added as the source of the alcohol. The lactic and alcoholic fermentations may be carried on simultaneously, but a smoother, creamier product is obtained if a cultured buttermilk is first prepared and the alcoholic fermentation carried out subsequently.

An active yeast culture is prepared by adding a teaspoonful of sugar and a cake of yeast to a pint bottle of water that has previously been boiled and cooled. The bottle is plugged with cotton or covered with a loosely-fitting cap and set in a warm place over night. Cultured buttermilk should be prepared as directed earlier in this chapter, careful attention being given to breaking up the curd in order to obtain a smooth product.

The quantity of sugar to be added to the buttermilk should be governed by the amount of carbon dioxide desired in the finished product. It should be sufficient to make the fermented milk effervescent and to impart the sharp taste of carbonated beverages, but too great a quantity will cause the liquid to blow out of the bottles when the stoppers are removed or may even cause the stoppers to blow out or the bottles to explode. From 1 to 1.5 percent of sugar will give the proper amount of gas.

After the addition of the sugar, the yeast culture is added in the proportion of 1 percent, the buttermilk thoroughly mixed and bottled. Only heavy bottles such as gingerale or pop bottles should be used. They should be carefully cleaned and either boiled in water or steamed before being used. After filling they should be capped with pop-bottle caps, or stoppered and the stoppers securely wired.

The bottled milk should be incubated for 3 to 5 days at 65 to 70°F. The bottles should be shaken as often as is necessary to keep the curd in a finely divided condition. At the end of the incubation period the milk should be effervescent when poured from the bottle, should be smooth and creamy, and should have the pleasant acid taste of buttermilk accentuated by the tang of the gas and the trace of alcohol. If the curd is coarse and contains large gas bubbles, or if the milk has a yeasty taste, the fermentation has been too vigorous and should have been carried out at a lower temperature. The bottles of fermented milk should be placed in a refrigerator, where the milk may be kept for a week or more without appreciable change.

**Preparation of Kefir Buttermilk.** A product closely resembling churned buttermilk may be prepared by the use of kefir grains as fermenting agent. Milk containing 2 to 3 percent fat is pasteurized and cooled to 70°F. About ¼-pound of active kefir grains should be used for each gallon of milk, and, for small-scale operation, they should be placed in a cheesecloth bag and submerged just below the surface of the milk. The milk should be held at 70°F until it coagulates and the acidity is about 1 percent. The bag of grains is then removed and the adhering cream scraped off. The fermented milk is cooled to 50°F, or lower, and the curd broken up by stirring as in the preparation of cultured buttermilk. It is then bottled and is ready for use immediately, or it may be stored in a refrigerator. The bag of grains should be washed in cold water and the excess water removed by gentle pressing. The grains may then be used to ferment another batch of milk.

In large-scale operation, the milk should be fermented in a vat and the grains scattered on the surface of a piece of cheesecloth that is of the size of the vat and is submerged 2 or 3 inches below the surface of the milk. At the end of the fermentation, the grains are removed in the cheesecloth and washed in preparation for re-use.

## YOGHURT[1,3,5,14]

The product known as yoghurt has characteristics intermediate between those of the common types of cultured milks and those of the soft cheeses. It is made by fermentation of concentrated whole milk, is snow-white in color, is acid in flavor, contains little or no alcohol, and has a fine, smooth texture and a firm body like custard or junket. It is conveniently eaten with a spoon.

Fermented milks of the yoghurt type have been made for many centuries, principally in the countries near the eastern end of the Mediterranean Sea. The dahi of India, the matzoon or mazun of Armenia, the kisselo mleko of the Balkan countries, the leben of Egypt, and the gioddu of Sardinia are all products similar to the yoghurt of Turkey. According to tradition, Abraham was the first man to make yoghurt, he having been taught the process by an angel.

Yoghurt has become popular in the countries of western Europe and is being made commercially in the United States. A liquid product very similar to Bulgarian cultured milk has been accepted in this country as yoghurt by people not familiar with the characteristics of the true type, and this has resulted in considerable confusion between these two different varieties of fermented milks.

Defined from the standpoint of its manufacture, yoghurt is a semi-solid product made from concentrated whole milk containing between 19 and 20 percent total solids and fermented by a mixed culture of three organisms, designated *Streptococcus thermophilus, Bacterium bulgaricum* and *Plocamobacterium yoghourtii.*

**Propagation of Cultures.** Authentic yoghurt cultures should be obtained from reliable firms that carry such cultures for sale. Mother cultures should be prepared and carried in duplicate and transfers made every two days. The cultures, at time of transfer, should have the normal flavor of yoghurt; abnormal cultures should be discarded and only normal cultures used for further transfer, or for the preparation of bulk starter. If both duplicates are abnormal, a new culture should be purchased.

Two wide-mouthed, half-pint bottles are filled to half or two-thirds capacity with fresh skim milk and fitted with cotton plugs. The bottles and contents are heated in a sterilizer for 15 minutes at 15 pounds pressure, or in steam or boiling water for an hour at atmospheric pressure. The bottles are cooled to 115°F and about 5 milliliters of inoculum added to each by methods of sterile technic. The cultures are then placed in an incubator at a temperature of 85 to 90°F for 48 hours, at the end of which time the milk should be firmly curdled. New transfers should then be made and the remainder of the cultures used in preparation of bulk starter for large scale production or directly in making yoghurt on a small scale.

For the preparation of bulk starter, a glass, glass-lined, or metal container with a loosely-fitting cover should be used. It should be of sufficient capacity to contain, when half or two-thirds filled, 2 parts of starter for each 100 parts of yoghurt to be made. The necessary quantity of skim milk is heated in the container at 180°F for 30 minutes, then cooled to 112°F, inoculated with 2 percent of the mother culture, well mixed, and allowed to incubate at 112°F for 3 hours or until curdled. It is then ready for use. If not to be used immediately, it should be cooled to about 65°F and, after 8 hours, to about 60°F. It should, however, be used as promptly as possible after curdling.

**Preparation of Yoghurt.** A concentrated milk containing between 19 and 20 percent total solids is prepared by evaporation of whole milk in a vacuum pan to two-thirds of its original volume, or by adding to whole milk either an equal weight of commercial evaporated whole milk or 7 percent of its weight of spray-dried whole milk, and mixing thoroughly. The concentrated milk is either flash-pasteurized or heated to 180°F for 30 minutes and then cooled to 115°F. Two percent of its weight of the bulk starter is added and the inoculated milk thoroughly mixed. After standing for about one and one-half hours, the milk is stirred for a few minutes and transferred to the containers in which it is to be marketed. These are usually one-half-pint, wide-mouth, glass jars provided with pasteboard caps. The containers of inoculated milk are incubated at 112 to 115°F for one and one-half hours longer, when the milk should be firmly coagulated. The jars of curdled milk are then placed in a refrigerator at 50°F, or lower. The yoghurt is ready for delivery to customers in about 8 hours. It will keep in good condition in a refrigerator for at least a week.

If an homogenizer is available, the concentrated milk may be homogenized before pasteurization and, after inoculation and mixing, transferred immediately to the glass jars and incubated for 3 hours. The purpose of this homogenization, or of the preliminary incubation in bulk for one and one-half hours, is to prevent the formation of a cream layer on the surface of the yoghurt.

## CHOCOLATE-FLAVORED MILK[2, 12, 15]

Chocolate-flavored milk is made by dairies in most sections of the United States, usually as a pasteurized, homogenized, chocolate-flavored skim milk or partially skimmed milk, and merchandised in ordinary milk bottles. Chocolate milk sterilized and sold in pop bottles is not as generally distributed. The manufacture of sterilized chocolate milk is described in Chapter 8.

**Ingredients.** Skim milk, partially skimmed milk or whole milk may be used as the basis of chocolate-flavored milk. Factors other than fat content being the same, the palatability of the beverage varies directly with the percentage of fat. Milk with 1 to 2 percent fat is most commonly used.

Many states and cities have regulations governing the manufacture, sale and labelling of chocolate milk. Some states permit the names "Chocolate Milk" and "Chocolate-Flavored Milk" to be used only on products made from whole milk, and, if skim milk or partially skimmed milk is used, permit only such designations as "Chocolate Drink" or "Dairy Drink." Local authorities should be consulted before attempts are made to sell these products.

Cocoa is the most widely used flavoring material, but liquor chocolate, which is usually considered to have a better flavor, can be used, although there are troublesome technical difficulties in its use that have not been overcome. Cocoas differ widely in their physical characteristics, the most pronounced differences being between the so-called Dutch-process and American-process cocoas. It is not necessary, however, to know the details of cocoa manufacture in order to select a suitable cocoa, since it will be judged on the basis of the flavor of the chocolate milk produced. Cocoa is used in the proportion of 1.0 to 1.5 percent, liquor chocolate in the proportion of 1.5 to 2.0 percent.

The percentage of sugar in chocolate milk is usually between 5 and 8. In proportioning sugar and chocolate, it should be borne in mind that a high degree of sweetness and strong flavor have a greater initial taste appeal than a low degree of sweetness and less flavor, but satisfy the appetite sooner and tend to become cloying.

Vanilla, 0.1 to 0.2 percent of salt, and spices in very small proportions may be added to enhance the flavor.

A stabilizer is usually added to delay or prevent settling of cocoa particles. It also aids in the prevention of cream rising. One percent of starch, gelatinized by heating in the milk to 190°F, 0.3 percent of gelatin, additional milk-solids-not-fat added as condensed skim milk, and 0.2 percent of sodium alginate are the stabilizing agents that have been most commonly used. In the past few years, carrageen, in a concentration of 0.04 percent, has been advocated as a stabilizer, but its scarcity during the war years limited its use. The presence of these substances increases the viscosity of the chocolate milk, which, if excessive, is undesirable, since a highly viscous product may entrap air bubbles or may have the appearance of having curdled and extruded whey. Although the prevention of settling of cocoa particles is considered to be due largely to the increase in viscosity produced by stabilizers, other factors are involved; for it has been shown that sodium alginate and carrageen effectively prevent sedimentation at a viscosity level lower than that necessary when other stabilizers are used.

**Processing.** The milk used in making chocolate-flavored milk is homogenized to prevent or delay the rising of cream. It may be homogenized after the addition of cocoa and sugar, but this has the effect of increasing sedimentation. After all the ingredients have been added, pasteurization is carried out for the usual sanitary reasons and to aid in the prevention of rising of cream.

Syrups containing cocoa, sugar and stabilizer or dry mixtures of cocoa and stabilizer may be purchased. Syrups are added to heated homogenized milk, and the mixture pasteurized, bottled and cooled. The dry mixes may be mixed with sugar before being added to the milk.

Syrups may be made in the dairy plant, if desired, in quantity sufficient for several days use, or the chocolate milk may be prepared each time by adding cocoa, sugar and stabilizer directly to the milk without the preliminary preparation of a syrup. Directions are given for both these procedures.

*Procedures.* For a syrup to be used in making a chocolate milk containing 1 percent of cocoa, 5 percent of sugar, and 0.2 percent of sodium alginate, these three ingredients are weighed out in the proportion given. The 0.2 part of alginate is mixed thoroughly with 5 times its weight of sugar, and the 1 part of cocoa similarly mixed with the remainder—4 parts—of the sugar. To the mixture of cocoa and sugar, 4 parts of sweet skim milk are slowly added with stirring and the syrup is stirred until smooth. It is then heated to 150°F and the mixture of alginate and sugar added gradually with stirring. The syrup is then heated to between 180 and 190°F, held for 15 minutes, then quickly cooled to below 50°F, and held at this temperature until used. If water is used instead of skim milk, the syrup will have better keeping quality, but there will be greater dilution of the non-fat solids in the finished chocolate milk. Greater proportions of cocoa and sugar may be used in the syrup, if more flavor and sweetness are wanted in the product. If liquor chocolate is used instead of cocoa, it is melted, thinned gradually with skim milk to a smooth paste, the alginate and sugar added to the hot paste, and the mixture heated and cooled as directed above.

To make the chocolate milk, 90 parts of sweet skim milk, partially skimmed milk, or whole milk, are heated to 140 to 145°F and homogenized at 1500 to 2000 pounds pressure. Ten parts of the syrup, prepared as described, are thoroughly mixed with the milk and the mixture is held at 145 to 150°F for 30 minutes. It is then cooled as rapidly as possible, bottled and held at a temperature below 50°F until used.

To prepare a chocolate milk of the composition given above without the preliminary preparation of a syrup, 1 part of cocoa previously mixed with an equal weight of sugar is added to 90 parts of homogenized milk and the mixture heated with stirring to 150°F. The 0.2 part of sodium alginate mixed with 5 times its weight of sugar is then added gradually and thoroughly stirred in. The remaining 3 parts of sugar (making altogether 5 parts of sugar) are then added, the mixture heated at 145 to 150°F for 30 minutes, cooled rapidly, bottled, and refrigerated below 50°F until used.

Carrageen in the proportion of 0.04 part may be substituted for the 0.2 part of alginate. It is claimed that 0.04 percent of carrageen is adequate to prevent settling of cocoa fiber and rise of fat and that this stabilizing effect is obtained without producing an objectionable thickening of the milk. Larger percentages of carrageen cause thickening and have, in some instances in the past, imparted an objectionable seaweed flavor. Carrageen free from seaweed flavor can now be obtained for use in chocolate milk. A process for stabilizing chocolate milk by means of a mixture of carrageen and alginate has been patented[9] and is claimed to give a more satisfactory product than one made with either stabilizer alone.

The use of a mixture of starch with agar or with other gums has been patented.[11] The chocolate milk contains between 1 and 3 percent starch and between 0.01 and 0.20 percent gum and is heated for from 20 to 30 minutes at 170 to 200°F to gelatinize the starch. Higher temperatures should be avoided, since they cause thickening of the milk by coagulating casein.

If it is necessary to hold chocolate milk for a long time or at room temperature, it should be sterilized in bottles or cans, as described in Chapter 8.

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# 3

# FERMENTATION PRODUCTS FROM WHEY

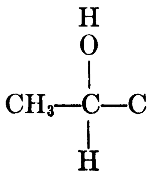
## INTRODUCTION

The substances that can be produced by fermentation of the lactose of whey are not unique, since they can be obtained by fermentation of other sugars. In general, the products of fermentation are determined primarily by the choice of fermenting organism rather than by the sugar in the medium, but the relative proportions of products may be altered in many instances by influences attributable to the composition of the medium or its physical characteristics. Many organisms that ferment other sugars do not ferment lactose; consequently, the chief factors determining whether lactose is to be the carbohydrate in a commercial fermentation are the availability of an organism that can convert lactose efficiently into the desired product and the cheapness of whey relative to that of other available sources of fermentable carbohydrate, such as cane, beet, or sorghum molasses, or corn sugar. The presence of certain essential nutrients in whey gives it an advantage in some instances, and the relative ease of purification of the product may be a favoring factor.

Many substances are reported to have been produced by fermentation of lactose. Those produced in yields sufficient to warrant consideration for commercial production from whey include ethyl[39] and butyl[1] alcohols, butyric,[3, 11, 12] citric,[50] acetic,[42] propionic,[43] and lactic[54] acids, acetylmethylcarbinol,[13] acetone, riboflavin, and yeast.[18] Lactic acid and riboflavin are produced commercially by fermentation of whey. Butyl alcohol and acetone are byproducts of the production of riboflavin. Lactose is used as an adjunct in the medium in which penicillin is produced. It seems unlikely that any of the other compounds listed above will be made commercially from whey, with the possible exceptions of yeast, ethyl alcohol, and acetic acid derived from this alcohol.

## LACTIC ACID AND LACTATES

Lactic acid, in the language of chemistry, is alphahydroxypropionic acid,



$CH_3\text{—}\underset{H}{\overset{OH}{C}}\text{—}COOH$. Because of the asymmetry of the alpha carbon atom, this acid exists in two modifications—a dextro acid and a levo acid. The commercial acid is a mixture of the two forms, usually in equal proportions, and it is therefore inactive in respect to rotation of polarized light. Hence, the confusing details of configuration and rotation of the active acids and their salts need not be discussed here.

Lactic acid is both an alcohol and an acid, and therefore its molecules can form esters with one another. In water solutions containing less than 20 percent of lactic acid, the acid is in the simple monomeric form, but solutions of greater concentration contain some of the acid in the form of esters involving two or more of the simple molecules. If a sufficiently concentrated solution of lactic acid is distilled under vacuum, the distillate consists largely of a dimeric double ester in which the acid and alcohol groups of one molecule have reacted with the alcohol and acid groups, respectively, of a second molecule. This ring compound is known as lactide.

Inasmuch as the heating of lactic acid solutions at atmospheric pressure evaporates water and causes the formation of esters of gradually increasing complexity and viscosity, lactic acid cannot be distilled or crystallized by ordinary means. This behavior gives rise to difficulties in the separation of lactic acid from other substances in the medium in which it is produced.

Lactic acid has been known for a long time as the principal acid in sour milk, but only since 1930 has it been produced commercially from milk byproducts. Until that time the few million pounds of lactic acid and lactates required annually in the United States were produced from molasses and corn sugar with *Lactobacillus delbrueckii* as the fermenting agent. *L. delbrueckii* (Bergey) does not ferment lactose; hence, a different organism, usually designated *L. bulgaricus*, is used in producing lactic acid from whey. Its activity is enhanced by the associative action of a mycoderm, which, though producing mainly alcohol when growing alone, produces only negligible quantities when growing in association with the so-called *L. bulgaricus*.

Whey, either from cheese or casein production, is the milk byproduct used in the manufacture of lactic acid. About 12 billion pounds of whey, containing 600 million pounds of lactose, is produced annually in the United States. Of this, about 2 billion pounds, containing 100 million pounds of lactose, is dried for use in foods and feeds, and 0.5 billion pounds, containing 25 million pounds of lactose, is used in the production of lactose. Thus, there is left 9.5 billion pounds of whey, containing 475 million pounds of lactose, potentially available for conversion into lactic acid or other fermentation products. From this 9.5 billion pounds of whey could be obtained 400 million pounds of lactic acid, which is approximately 100 times the quantity of lactic acid produced in this country annually.

Lactic acid is marketed in four principal grades:—technical, as 22 or 44 percent dark; edible, as 44 percent light or 50 percent water-white; U.S.P.

as 85 percent; and plastic, as acid in concentrations not less than 50 percent. The technical grade receives little or no refining treatment and is used mainly in the production of leather to neutralize the lime used in the processing of hides. For this purpose a weak organic acid is needed which forms a water-soluble salt with the calcium of the lime, as does lactic acid. For edible lactic acid there are no generally accepted requirements other than those for food components in general. Buyers of edible acid, however, usually set up certain specifications regarding color, odor and flavor. Edible lactic acid is used in pickle manufacture and, especially when citric acid is scarce, as an acidulant in bottled drinks and sherbets. Lactic acid of the grade specified by the United States Pharmacopoeia[37] contains not less than 85 nor more than 90 percent lactic acid, not more than 6 milligrams ash per 5 cubic centimeters, and not more than 10 parts heavy metals per million. It gives negative results when tested for sugars or matter carbonizable by sulfuric acid, and no turbidity when tested with the usual precipitants for chlorides, sulfates, citrates, oxalates, phosphates, or tartrates. Lactic acid of plastic grade is used in the manufacture of transparent cast phenolic resins. For such use the acid must meet such specifications as: that the 50-percent acid shall contain not more than 5 parts of chlorine or 50 parts sulfate per million; not more than 0.05 percent of ash or a trace of iron; and shall meet rigid requirements as to freedom from color. Only comparatively recently has there been produced domestic acid that meets the requirement for the plastic grade.

Lactic acid is used to a comparatively minor extent in the form of its salts, such as the lactates of calcium, sodium, potassium, iron and copper. According to the United States Pharmacopoeia,[37] calcium lactate for pharmaceutical use contains not less than 25 nor more than 30 percent water and, after being rendered anhydrous by drying to constant weight at 120°C, contains not less than 98 percent of $(CH_3 \cdot CHOH \cdot COO)_2Ca$; 1 gram in 20 cubic centimeters of distilled water requires not more than 0.5 cubic centimeters of 0.1 normal sodium hydroxide to render the solution neutral to phenolphthalein; it contains not more than 15 parts heavy metals per million and no volatile fatty acids; and 1 gram yields not more than 5 milligrams of sulfates of magnesium and alkali metals when treated with sulfuric acid. Sodium lactate is used as a substitute for glycerol in the textile industry. A mixture of sodium and potassium lactates has been used as a fire retardant.

Ethyl, butyl and amyl lactates are used in the lacquer industry as solvents for cellulose derivatives. Methyl lactate has been advocated as an intermediate for the industrial production of acrylates; and as the chief constituent of highly resistant coatings for metal.[9, 48, 49]

It would probably not be profitable to make lactic acid on a scale requiring less than 100,000 pounds of whey per day. If the crude calcium lactate produced were marketed without conversion to lactic acid or other lactates requiring refining operations, less equipment and less highly technical labor would be needed and it is possible that operations on a slightly smaller scale might be practical. Only a careful technical and economic survey could determine the advisability of operation on such a limited scale.

**Manufacturing Equipment.** The equipment[7, 36] required for producing calcium lactate from whey is shown in the accompanying flow sheet, Figure 3. The culture vats should be glass-lined, be jacketed for use of steam in heating, and have a capacity of 700 gallons each. The fermentation vats may be of wood, preferably cypress, and should be fitted with agitators and steam coils. The capacity of each should be 7000 gallons. The filter press may have either iron or wooden plates and frames, suitable inner dimensions of the frames being 1 x 36 x 36 inches. A 7000-gallon vat similar to the fermentation vats is suitable for storage and processing of the fermented and clarified whey. The vacuum evaporators used for concentrating the fermented whey may be of copper or stainless-steel construction. The crystallizers are jacketed boxes lined with stainless-steel and provided with heavy casters for moving them about. Connections on the jackets are provided for cooling water and outflow. The capacity of each crystallizer should be about 300 gallons. The centrifugal should have its basket of stainless-steel. If the calcium lactate is to be dried, a tunnel drier is required, such as is described in Chapter 10 for drying casein.

For refining calcium lactate and converting it into lactates of other metals, a jacketed, stainless-steel or glass-lined vat and a wooden filter press are needed, their sizes depending on the scale of operation. The evaporator used in these operations should be preferably of stainless steel.

For conversion of calcium lactate to lactic acid, a jacketed glass-lined vat, a filter press having hard-rubber plates and frames, and a vacuum filter of chemical stoneware are necessary. Lactic acid in concentrations as great as 50 percent can be made without evaporating water from lactic acid solutions, but if acid of greater concentration is made, an evaporator of highly corrosion-resistant metal is required.

**Processes of Manufacture.**[7, 20, 36, 51] Whether the product is to be lactic acid, a metallic lactate, or a lactic ester, calcium lactate is usually made as an intermediate substance. The reasons for this are that: (a) hydrated lime is a comparatively cheap material to use for the neutralization of the acid during its formation; (b) calcium lactate can be purified by recrystallization; and, above all, (c) soluble lactates or lactic acid can readily be prepared in a relatively pure condition from calcium lactate by reaction with

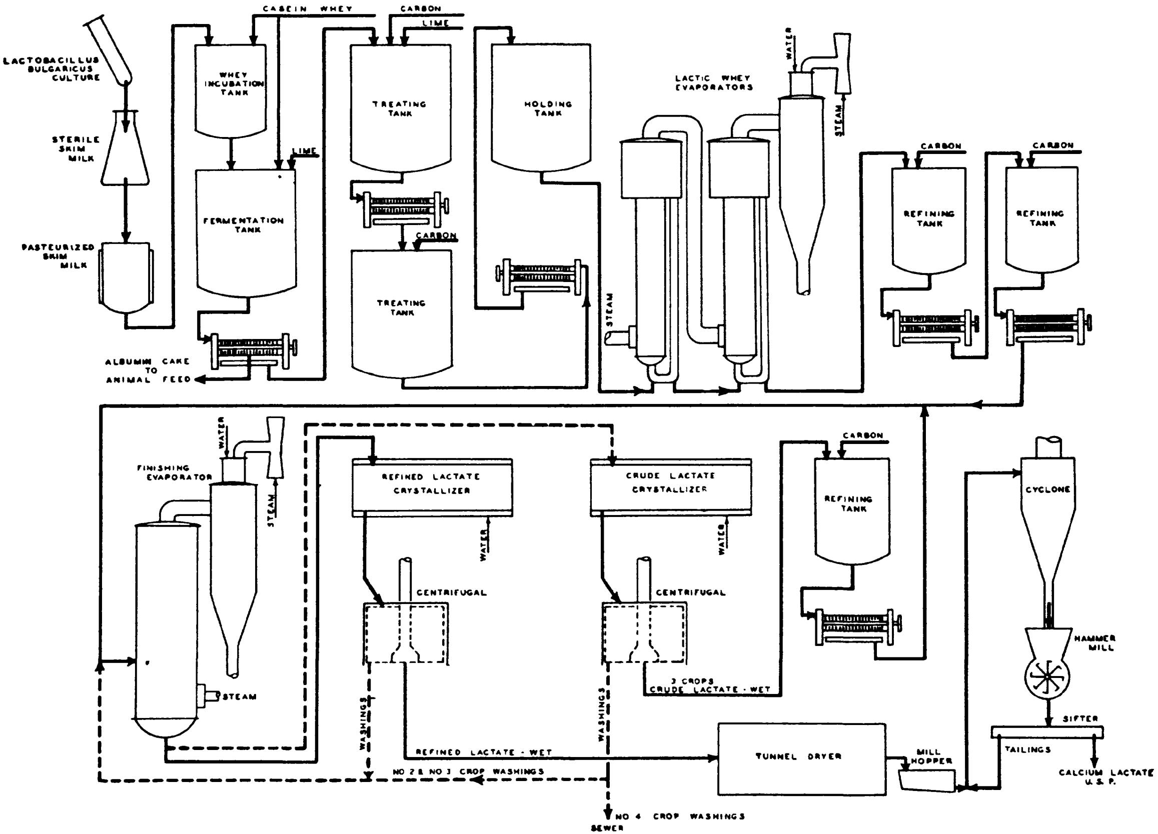


Fig. 3. Process for the manufacture of calcium lactate from whey. (*Courtesy of Chemurgic Division, Sheffield Farms Company, Inc., Subsidiary of National Dairy Products Corporation.*)

the proper metal sulfates or sulfuric acid, since calcium sulfate is practically insoluble in water and hence can readily be separated from the product by filtration. Making soluble lactates, as, for example, sodium lactate, by using sodium hydroxide or carbonate as the neutralizing agent during fermentation, is practical, provided contamination by the salts present in the whey is not objectionable. The processes of manufacture are conveniently divided into the fermentation of whey to produce a solution of calcium lactate, purification of calcium lactate solution, and conversion of calcium lactate into other lactates and lactic acid.

*Fermentation.* The culture employed commonly for the conversion of lactose to lactic acid is a mixed culture of a lactobacillus of the bulgaricus type and a mycoderm. It is known among dairy bacteriologists under the designation of "ga" and is Culture No. 9223 in the American Type Culture Collection. Stock cultures are carried in bacteriological laboratories in sterilized skim milk, but in a plant laboratory skim milk heated to boiling for 30 minutes or even pasteurized skim milk may be used, since possible contaminating organisms grow very poorly at the reactions and temperatures of the cultures. The cultures are incubated at 110°F for 24 hours between successive transfers.

A laboratory flask of culture is used as inoculum for each of two 10-gallon cans of pasteurized skim milk. After 24 hours of incubation at 110°F, the contents of each can is used as inoculum for 500 gallons of sterilized whey contained in a 700-gallon, glass-lined, jacketed culture vat. After 24 hours of incubation, the contents of two culture vats are added to 4000 gallons of raw whey in a 7000-gallon fermentation vat. The temperature of the whey should be brought to 110°F before addition of the culture. The contents of the vat are mixed and the temperature of the whey maintained at 110°F during the fermentation period. Both in the culture vats and in the fermenters, the surface of the fermenting liquid should be exposed to air in order that the growth of the mycoderm may be favored by the presence of oxygen and by a slightly lowered temperature at the surface.

It is necessary from time to time to neutralize the lactic acid being formed during the fermentation; otherwise, the fermentation would decrease in rate and eventually stop. Approximately 1000 pounds of hydrated lime are required for the neutralization of each batch and it is essential that it be free of magnesia. The lime should be added in the form of a slurry and thoroughly mixed with the whey by adequate agitation after each addition. Liming should be done every four to six hours, the quantity added being controlled by determinations of acidity, or, better, the pH value. The reaction of the whey should be kept between pH 5.0 and 6.0. With acidity determinations as controls, liming should reduce the acidity to about 0.3 percent lactic acid.

The approach to completion of the fermentation is indicated by a decreasing requirement for lime, but it is advisable to test for residual sugar by means of Fehling's solution. As soon as the sugar is practically all fermented, which should require about 42 hours, the whey is neutralized with lime to approximately pH 7.0, or 0.1 percent acid, and heated to the boiling point. The heating is continued until the whey protein is completely coagulated, and then discontinued to allow the coagulum to settle.

*Purification of calcium lactate.*[7, 51] The supernatant liquid is decanted to a storage vat and the sludge filtered in a filter press, the filtrate being run to the storage vat. For a slightly better product the supernatant liquid may be filtered also, but it is preferable to filter the supernatant liquid and the sludge separately to avoid filtration difficulties that arise if they are handled together at this stage. The press cake, which is largely protein, may be dried, and marketed as a stock feed.

If crude calcium lactate is to be made, the clarified solution is concentrated in a vacuum evaporator to 15° Bé., allowed to crystallize, and the crystals centrifuged and dried. If crude 22 percent lactic acid is to be made, the solution is evaporated to 20° Bé. before conversion to acid. These operations are carried out in the same manner as with the purified calcium lactate solution and are described in subsequent paragraphs.

For the production of the refined grades of calcium lactate and lactic acid, the solution should be decolorized and further clarified before concentrating. A small percentage of vegetable carbon is added, the quantity depending on the activity of the carbon, and the solution is heated to boiling and maintained at this temperature for 15 minutes. Heating is then discontinued and, with the agitator running, hydrated lime is added slowly until the reaction of the liquid is approximately pH 10. Stirring is continued until a sample taken from the vat separates sharply into a grayish precipitate and a clear supernatant liquid. The batch is then filtered in a filter press, yielding a clear, water-white solution of calcium lactate. Lactic acid, if available, is added to the solution until its reaction is approximately 7.0, or its acidity is about 0.05 percent. Sulfuric acid may be used for this purpose, but, if it is used, a filtration is necessary for removal of the calcium sulfate formed in the neutralization.

The neutral calcium lactate solution is evaporated to 15° Bé. and pumped to the crystallizers. Cold water is circulated in the jackets of the crystallizers until no further crystallization takes place, which will require 10 to 12 hours. The wet crystalline mass is shoveled into the basket of the centrifugal, in which the mother liquor is spun out and the crystals washed several times with small quantities of cold water. The crystals may then be either dried, and marketed as technical calcium lactate, or dissolved in hot water, the solution treated with activated carbon, filtered, evaporated and crystallized, and the crystals centrifuged, washed and dried, and marketed as U.S.P. calcium lactate. The drying of calcium lactate is carried out in a tunnel drier below 212°F in a current of filtered air. The dry crystals are packed in barrels of 200 pounds capacity.

*Production of other metallic lactates.*[51] Calcium lactate may be converted into lactates of other metals, such as those of ammonium, sodium, potassium, magnesium, iron and copper, by mixing solutions of equivalent quantities of calcium lactate and the sulfate of the metal, the lactate of which is desired. In making ammonium, sodium or potassium lactate, the corresponding carbonate may be used instead of the sulfate. As the source of calcium lactate, the decolorized solution of calcium lactate that has not been crystallized, a solution of crystals of the technical grade, or a solution of the refined crystals may be used. Obviously, the purer the calcium lactate used, the purer the product obtained.

Quantities of solutions of the two reacting substances are mixed in proportions calculated to be equivalent. The mixture is stirred and the filtrate from a small filtered sample is divided and tested for excess calcium lactate by means of ammonium oxalate solution, and for excess sulfate (or carbonate) by means of barium chloride solution. More of the needed reactant is added, the mixture stirred and tested, and this repeated until the components are properly balanced. The precipitate is removed in a filter press and the filtrate concentrated. Since ammonium, sodium and potassium lactates are not readily crystallized, they are marketed as syrupy solutions; the lactates of most other metals can be crystallized.

Metal lactates can be prepared also by causing lactic acid to react with the hydroxide or carbonate of the metal. This involves one more step than the process just described, since the lactic acid must first be prepared from calcium lactate and then neutralized with the metal hydroxide or carbonate.

The process of Robinson[38] is directed to the production, from sweet whey, of a solution of sodium lactate suitable for use as an emulsifying agent in the making of processed cheese. After residual fat has been removed by passage through a cream separator, the sweet whey is run to a fermentation vat and the lactose fermented either by the lactic organisms normally present or by cultures added for the purpose. If the fermenting organisms are lactic streptococci, the whey is incubated at 80°F; if lactobacilli are used, the whey is incubated at about 105°F. The fermentation is allowed to proceed for 3 or 4 days, which is sufficient time for substantially complete conversion of the lactose into lactic acid. Sodium hydroxide or sodium carbonate is added at least every 12 hours during the fermentation period in quantities sufficient to bring the reaction into the most favorable pH range. When the fermentation is complete, the reaction is brought to neutrality and the whey fed to a vacuum evaporator in which it is concentrated at a temperature not exceeding 160°F. A voluminous flocculent precipitate of whey protein forms in the later stages of concentration, and, when a filtered sample gives no precipitate on further evaporation, the concentrated whey is filtered. The syrupy filtrate may be used without further concentration, or it may be concentrated until it contains 65 percent of sodium lactate. The filtration may be omitted, if preferred, since the presence of whey protein in cheese products is not objectionable.

*Production of lactic acid.*[7, 51] Lactic acid is prepared from a solution of calcium lactate as produced in the fermentation or after any of the steps of refining. The dark technical grade can be made from a calcium lactate solution that has had no decolorizing treatment, and a lighter grade from the solution after decolorization. The edible and U.S.P. acids usually are made from calcium lactate that has been crystallized at least once. Less refining of the lactic acid is needed, the better the grade of calcium lactate used. All surfaces in contact with lactic acid should be of glass, rubber, or acid-resistant metal.

The solution of calcium lactate used should be of a concentration of approximately 20° Bé. If fermentation liquor is used, it should be concentrated to 20° Bé. and kept warm until the sulfuric acid is added; if crystals are used, they should be dissolved in sufficient warm water to give a solution of this density. If solutions of this density are allowed to cool, they are likely to set in hard masses that are difficult to handle. Sulfuric acid of 66° Bé. concentration is run slowly into the warm, agitated solution of calcium lactate until tests on filtered samples show that equivalence has been reached. Excess calcium lactate can be tested for by means of ammonium oxalate solution; excess sulfuric acid by means of barium chloride solution. The calcium sulfate is removed by filtration in a filter press equipped with rubber plates and frames, and washed with water, the washings being kept separate from the filtrate and used to dissolve calcium lactate in a subsequent operation or to dilute the lactic acid solution to 22 percent, if that final concentration is desired. By following the directions given, a solution containing somewhat more than 22 percent of acid should be obtained. This may either be diluted to 22 percent or be used in making acid of greater concentration, as described below.

In making acids of 44 percent or 50 percent concentration, crystals of calcium lactate are dissolved in the lactic acid made as described above, sulfuric acid added as before, and the calcium sulfate removed by filtration. This should give an acid of 35 to 40 percent concentration. By one more "building up" operation, the concentration of the acid can be increased to a percentage greater than the 44 or 50 desired. It may then be reduced to exactly 44 or 50 percent by addition of water or, preferably, the dilute acid obtained by washing the press cake of calcium sulfate. It should be obvious that it is not necessary to balance the acid and lactate precisely until the last stage of a "building up" operation. More highly concentrated lactic acid, such as 85 percent, is obtained by evaporation of water from approximately 50 percent acid in an acid-resistant vacuum evaporator. Decolorization and removal of heavy metals should precede this evaporation.

*Purification of lactic acid.* Lactic acid prepared as above described is appreciably colored. To obtain a colorless acid, it is necessary to treat with carbon, and, to prevent subsequent development of color, it is necessary to remove heavy metals, particularly iron, by precipitation with a ferrocyanide. These operations may be carried out on the 22, 44, or 50 percent acid. The quantity of potassium ferrocyanide solution to be added is determined by experience or by a laboratory test on a sample of the acid. The quantity should be sufficient to remove all the heavy metal present, but excess should be avoided as far as possible, since cyanide-like odors are likely to appear in acid containing ferrocyanide. Ferric ferrocyanide is recognized by its blue color; copper ferrocyanide is brown. After the addition of the potassium ferrocyanide solution, a small percentage of activated vegetable carbon is added, and the solution heated nearly to boiling. The flocculated mixture of carbon and heavy-metal ferrocyanides is removed by filtration in a filter press equipped with hard rubber plates and frames. The filtered acid should be water-white and should not develop color during the evaporation necessary to produce acid of greater concentration. The washings are combined with other acid of like concentration.

Several other methods have been devised for the refining of lactic acid and on most of these methods patents are still in force. One of the earlier procedures consists of preparing the zinc salt, as by the reaction of lactic acid and zinc carbonate or oxide, purifying it by recrystallization, and converting it to lactic acid by means of hydrogen sulfide. The zinc sulfide formed is removed by filtration, and the excess hydrogen sulfide by aeration. A procedure of this type is part of a patent.[4]

The conversion of magnesium lactate to lactic acid by means of sulfuric acid in an organic solvent in which the resulting magnesium sulfate is insoluble has been advocated,[17, 40] but apparently not used commercially.

The use of silver compounds to remove cyanides has been patented.[16]

Several patents have been issued on methods of oxidizing impurities by means of oxidizing agents such as ozone, hydrogen peroxide and nitric acid.[14, 15, 30, 46, 47] Methods of distillation usually involving vacuum or steam under pressure are advocated for use in connection with these oxidative procedures.[14, 23, 29, 30, 46]

Extraction of lactic acid solutions by isopropyl ether, followed by extraction of the ether solution by distilled water,[19] has had commercial use.

An earlier method of purification involving ester hydrolysis has been revived[44] and has evoked industrial interest.[41, 52, 53] It consists of converting impure lactic acid into a lactic ester, such as methyl lactate, by esterification with an alcohol, distilling the ester to purify it, hydrolyzing the ester to lactic acid and the alcohol by means of distilled water, and removing the alcohol and excess water by distillation. This procedure avoids crystallization and filtration procedures and use of carbon and ferrocyanide and yields an acid of plastic grade.

*Other methods of manufacture.* Lactates have been made by synthetic methods from acetaldehyde and other starting materials, but such methods are more expensive than fermentation procedures. However, a chemical procedure for converting sugars, including lactose, into lactates may eventually compete with the fermentation process. This method involves the reaction, in relatively high concentration, of alkalies, such as sodium or calcium hydroxide, with sugars.[4, 28, 56, 58] The reaction may be carried out under pressure. Various other products are formed in the process, but yields of lactic acid as great as 67 percent of the theoretical have been claimed.

## RIBOFLAVIN

The demand for riboflavin for use in fortification of foods and in preparation of vitamin concentrates for therapeutic use was met earlier by isolation of riboflavin from rich natural sources, and more recently by its preparation by chemical synthesis. Many microorganisms synthesize riboflavin for their own metabolic requirements; under controlled conditions, some of the yeasts and clostridia can be made to produce it in quantities sufficient to make their synthetic activity the basis for commercial production of this vitamin. A process for producing riboflavin in a medium containing hexose sugars by means of the yeast *Candida guilliermondia* has been patented.[6] *Clostridium acetobutylicum* (Weizmann) can be made to produce sufficient riboflavin in whey so that the dried product will contain 90 grams per 100 pounds (1980 micrograms per gram) instead of 1 gram per 100 pounds (22 micrograms per gram), which is the proportion in dried unfermented whey. During World War II, the production of dried whey enriched by such a fermentation was undertaken commercially to furnish a product for use in feed for chickens, which require riboflavin for adequate growth and production of hatchable eggs. One-half pound of a dried enriched whey, standardized to contain 35 grams riboflavin per 100 pounds (770 micrograms per gram), if added to 100 pounds of flour, would supply 1.8 milligrams riboflavin per pound of flour, which is considered a suitable degree of enrichment of flour for bread-making. Riboflavin isolated from whey in crystalline condition or as an adsorbate on lactose is suitable for therapeutic uses.

**Clostridium Fermentation of Whey.**[1, 27, 31, 32, 57] Culture No. 4259 of the American Type Culture Collection is a suitable strain of *Clostridium acetobutylicum* (Weizmann) for the production of riboflavin in whey. It is first transferred to a sterile medium, such as liver extract, and heat-shocked, after which it is allowed to grow for a few hours in an incubator at 100°F. The culture is then added to a batch of sterile whey, the whey incubated at 100°F, and by repeated transfers the culture is increased until about 50 pounds is available for each 1000 pounds of whey to be fermented.

The iron content of the whey is adjusted, either by the addition of a ferrous salt or by addition of whey containing a known percentage of iron, so that between 1.5 and 2.0 parts of iron are present per million of whey. It is highly important that this adjustment be made, since either a less or a greater proportion of iron than that specified causes lowered yields of riboflavin.[1] Traces of strontium, tin, manganese, lithium and zinc should preferably be added, but are not essential. The reaction of the whey should be adjusted to between pH 6.0 and 7.0 by means of addition of sodium, potassium or calcium hydroxide, and 1 to 3 pounds of calcium carbonate should be added as buffer for each 1000 pounds of whey.

The whey medium is then sterilized by heating at 250°F for 15 to 20 minutes in a pressure vat lined with material that will not alter the iron content of the whey. After the whey has been cooled to 100°F, 50 pounds of culture is added for each 1000 pounds of whey and incubation is continued at that temperature until there is no noticeable increase in riboflavin content, which should be after 12 to 48 hours. It is important that the temperature of the whey be kept as close to 100°F as possible, since variations of as little as 5° higher or lower will decrease the yield of riboflavin.

Hydrogen and carbon dioxide formed during the fermentation are allowed to escape through a suitable vent. The ethyl and butyl alcohols and acetone formed, which have value as solvents, are distilled and recovered during the concentration of the fermented whey.

The whey, after partial concentration by evaporation of water and the volatile solvents, is dried by methods described in detail in Chapter 5. The dried whey is standardized to the desired riboflavin content by mixing with dried whey prepared from unfermented fluid whey.

**Methods of Concentrating Riboflavin.** Dilute aqueous solutions of riboflavin may be concentrated by evaporation of water, preferably in a vacuum evaporator. When separation from non-volatile impurities is necessary, which is usually the case, the riboflavin may be adsorbed on fuller's earth, the adsorbate washed with water, and the riboflavin released by elution with an alkaline solvent. Adsorption of riboflavin on lactose may be employed as means of both purification and concentration; this method is especially suitable when whey is the medium containing the riboflavin. Removal of lactose from whey by fermenting it to ethyl alcohol before evaporation has also been employed in concentrating riboflavin. Extraction with organic solvents may be used as a means of purification and is particularly desirable when the riboflavin is to be obtained as crystals.

*Method of Myers and Weisberg.*[33] The fermentation of lactose to alcohol may be applied to skim milk for the production of a de-lactosed skim milk for use in ice cream, or it may be applied to whey as a step in the concentration of the vitamins of the whey. After fermentation by means of lactose-fermenting yeasts, as described later in this chapter, the alcohol is recovered and the residue condensed or dried. A dried whey concentrate made in this way will contain about 30 percent protein and 30 percent salts. The vitamin potency can be greatly increased by extracting the dried whey concentrate with an alcohol and evaporating the alcohol from the extract. If an alcohol that is immiscible with water is used for this extraction, the extract may be re-extracted with water and the water removed from this second extract by evaporation; thereby, a still more highly concentrated vitamin preparation is obtained.

*Method of Dickens.*[8] This process increases the concentration of the vitamins in whey somewhat more than 20 times. Whey is concentrated in the ratio of 10 to 1 under vacuum at a temperature between 115° and 185°F. Sufficient hydrated lime, sodium hydroxide or sodium carbonate to give the concentrate a reaction of between pH 6 and 7 is then added, and the neutralized liquid is allowed to stand over night. In the morning the clear supernatant liquid is decanted, the sludge filtered, and the filtrate combined with the decanted solution. To this clear liquid is added from one-fourth to one-half its volume of 95 percent ethyl alcohol, the mixture allowed to stand for about 4 hours and then filtered. The filtrate is evaporated to about half its volume under vacuum at 115 to 185°F. This removes most of the alcohol, which may be recovered for re-use. The concentrate is allowed to stand in a refrigerator at about 50°F for several days, after which it is again filtered. The filtrate is then bottled and heated for an hour at 150°F on two successive days.

*Adsorption of riboflavin on fuller's earth.* The quantity of fuller's earth required for adsorption of riboflavin from any solution should be determined experimentally on a small sample taken from the solution. The solution should have an acid reaction, at least below pH 6.0 and preferably about pH 3.0. Sulfuric acid may be added, if necessary, to produce the acid reaction. Five grams of fuller's earth per 100 grams of riboflavin solution is suggested for experimental trial. The suspension should be stirred and the adsorption should be complete in about 10 minutes. The mixture is then filtered. If the filtrate contains riboflavin, as shown by its color, a larger proportion of fuller's earth should be tried. As soon as the requisite proportion of fuller's earth has been determined, the main batch of solution should be treated as described above. After the adsorbate is filtered, if should be washed several times with neutral or slightly acid water. It is then suspended in a conveniently small volume of 0.2 percent sodium hydroxide solution, the mixture stirred vigorously for 15 minutes, and centrifuged. Sulfuric acid is added to the filtrate until it is barely acid to methyl red indicator, and the solution concentrated to the desired degree in a vacuum evaporator. Light should be excluded insofar as possible in all operations involving riboflavin.

*Adsorption oj riboflavin on lactose.*[24, 25, 26] So many complicating factors are involved in the preparation of adsorbates of riboflavin on lactose that it is not possible to give precise directions for a procedure that will be applicable under all conditions. With an understanding of the principal factors affecting the adsorption and a knowledge of the solubility of lactose and of riboflavin, it should be easy to adapt the procedure outlined below to the preparation of an adsorbate of any desired composition from either an enriched or an unenriched whey.

The *quantity* of riboflavin that will be adsorbed depends, not only on the quantity of riboflavin present in solution, but also on temperature and on the percentage of lactose remaining in solution; so that, for high efficiency of adsorption, comparatively low temperatures and long times of crystallization are required. Furthermore, riboflavin exerts a retarding action on the crystallization of lactose, an effect that is greater, the greater the concentration of riboflavin in the solution and the less the degree of supersaturation with respect to lactose. The efficiency of adsorption may be high, but, if the degree of supersaturation with respect to lactose is relatively great, the *concentration* of riboflavin on the lactose will be small. If a high concentration of riboflavin in the adsorbate is desired, it is necessary to adsorb a relatively large quantity of riboflavin on the relatively small quantity of lactose crystallizing from a solution only moderately supersaturated with lactose and to conduct the crystallization at a comparatively low temperature. The excessive time required for complete crystallization under these conditions makes it unpractical to make adsorbates containing more than 300 micrograms of riboflavin per gram of lactose.

Two distinct operations are necessary for the production of an adsorbate of a definite concentration—the adjustment of the relative concentrations of lactose and riboflavin in the solution, and the crystallization of the adsorbate from the sufficiently concentrated, adjusted solution. The adjustment of relative concentrations usually requires the removal of lactose from the solution, but may, in some instances, require addition of lactose. If it is desired, for example, to produce an adsorbate containing 300 micrograms riboflavin per gram of lactose, the solution from which this adsorbate is to be crystallized should contain approximately 204 grams of lactose and 28 milligrams of riboflavin per liter. From each liter of such a solution, cooled to 40°F, complete crystallization will yield 84 grams of lactose containing slightly more than 25 milligrams of riboflavin. There will remain in solution 120 grams of lactose and slightly more than 2.5 milligrams of riboflavin per liter. If the clarified whey used as a starting material contains lactose in excess of the ratio required, it should be condensed to such a degree that, when the condensed whey is allowed to crystallize at 140°F, sufficient lactose will separate so that the ratio of lactose to riboflavin in the mother liquor will be that required for the adsorption operation. Very little riboflavin will be adsorbed under these conditions. The necessary calculations must be made on the basis of the analysis of the whey and the solubility of lactose at 140°F. After condensing in a vacuum evaporator, the whey should be allowed to crystallize at 140°F for 4 to 5 hours and then centrifuged. The crystals may be washed lightly and converted into refined lactose by methods described in Chapter 11. The filtrate should be condensed to the degree required to give a concentration of 204 grams of lactose and 28 milligrams of riboflavin per liter, transferred to a crystallizing vat, cooled to 40°F, seeded with lactose crystals, and stirred until crystallization is complete. The crystals should be separated from the mother liquor by filtration, preferably in a centrifugal, and dried in a tunnel drier at 150°F. The filtrate may be concentrated for the production of more adsorbate or for addition to vitamin feeds.

*Purification of riboflavin by solvent extraction.*[22] Extraction by means of organic solvents is used when the product desired is crystalline riboflavin. This procedure may be applied advantageously to concentrates of fermentation residues containing approximately 1000 micrograms of riboflavin per gram mixed with considerable amounts of contaminating material. The process consists essentially of successive extractions of riboflavin from aqueous solution by butanol and from mixtures of butanol and petroleum ether by water, the cycle being repeated several times, followed by a crystallization of the riboflavin from a mixture of water and acetone. A small-scale 2-cycle extraction procedure is outlined.

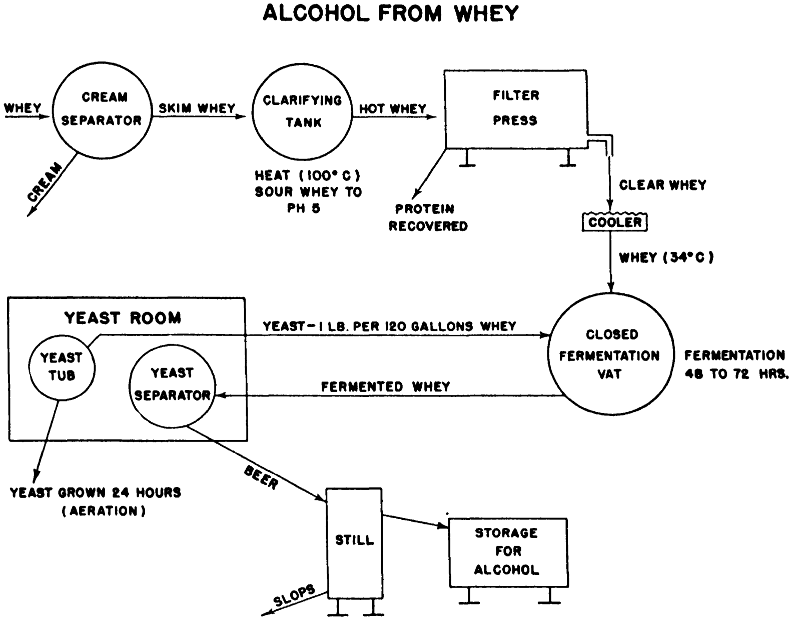
Clarified fermentation residue sufficiently concentrated so that it contains approximately 1000 micrograms of riboflavin per gram is used as starting material. Four liters are extracted six times with 3-liter portions of butanol. The 18 liters of combined extracts are reduced by evaporation in vacuum to 2.5 liters. The concentrated extract is mixed with 7.5 liters of petroleum ether and the mixture is extracted six times with 1-liter portions of water. The 6 liters of water extract are concentrated in vacuum to 700 cubic centimeters and the concentrate extracted five times with 500 cubic-centimeter portions of butanol. The 2.5 liters of butanol extract are mixed with 6.25 liters of petroleum ether and the mixture extracted five times with 500-cubic-centimeter portions of water. The combined water extracts are reduced to approximately 160 cubic centimeters by vacuum evaporation and, while the concentrate is still hot, 1.6 liters of hot acetone is added to it. This mixture is cooled to 32°F and maintained at that temperature for 5 hours. The supernatant liquid is decanted and the gummy residue extracted three times with 100 cubic centimeters of boiling acetone. The extracts and the supernatant solutions are combined and concentrated to 70 cubic centimeters by evaporation in vacuum. To the hot extract is added 70 cubic centimeters of acetone and the mixture is maintained at 32 to 40°F for about 16 hours. The crystallized riboflavin is removed from the liquid and washed, first with a mixture of equal volumes of water and acetone, and then with acetone alone. It is then dried in a vacuum. The riboflavin should be protected from light throughout the process, and the product should be packaged in light-proof containers.

## ETHYL ALCOHOL[2, 5, 10, 21, 34, 35, 39, 42]

For the production of ethyl alcohol by fermentation of the lactose of whey, it is necessary to employ a special lactose-fermenting yeast, since yeasts of the *Cerevisiae* type, commonly used for fermentation of glucose and sucrose, do not ferment lactose unless it has been previously hydrolyzed. Several lactose-fermenting yeasts have been found to give reasonably high yields of alcohol. *Torula cremoris* (American Type Culture Collection No. 2512) is recommended, since it has been shown to be capable of producing alcohol from lactose in yields of as much as 80 percent of the theoretical.

**Propagation of the Culture.** In preparation for the fermentation, the yeast culture is propagated until about 6 pounds of yeast (wet) is available for each 1000 pounds of whey. The test-tube culture is added to about 100 cubic centimeters of sterilized whey and the mixture incubated for 3 to 4 days at 100°F, with air constantly bubbling through. This culture is then added to about 500 cubic centimeters of sterilized whey and incubation carried out as before. By repeating the transfer to increasing quantities of whey, the culture is built up to give the required amount of yeast, with the modification that the last transfer is to diluted whey containing approximately 1.5 percent of lactose—1 part whey to 2 parts water—and this dilute culture is incubated only 24 hours.

**Fermentation.** The whey to be fermented is adjusted to a reaction of about pH 4.5 by addition of sulfuric acid, heated to boiling, filter-aid added, and the whey clarified by filtration. The clarified whey is then cooled to 100°F. Two ounces of ammonium sulfate and a culture containing about 6 pounds of yeast are added for each 1000 pounds of whey, and the fermentation carried out at 100°F. About 24 hours are required for completion of the fermentation, which is attained when the Brix reading is about 1.3° or a saccharimeter reading shows a slight negative rotation.



.Fig. 4. Manufacture of ethyl alcohol from whey.

**Refining.** The fermented whey should be filtered in a centrifugal to remove the yeast. The filtered whey is then distilled until about 25 percent of the original volume has come over. Foaming during distillation can be decreased by adding a small quantity of hydrogenated fat to the still pot. The distillate should contain about 14 percent alcohol by volume.

If it is desired to convert the alcohol to spirit vinegar, it is diluted to about 13 percent by volume, a small percentage of malt added, the mixture put through a vinegar generator, and the product filtered and standardized to contain the customary percentage of acetic acid.

## FOOD YEAST

Interest in yeast as a source of dietary protein has caused a number of investigators, chiefly in Europe, to study the possibilities of utilizing whey as a medium for yeast production. There appears to have been no commercial development based on these studies as yet. It has been found desirable to condition the whey prior to fermentation.[59] For example, the growth rate of *Saccharomyces fragilis* was found to be 50 percent greater in boiled and filtered whey than in untreated whey,[10] and acidification of diluted whey to pH 1.0–4.0 before sterilization, followed by adjustment to pH 5.0–8.0 before inoculation, increased the yield of yeast solids.[18] Yields of approximately 40 grams of yeast, containing 14 grams of protein, have been obtained from 100 grams of lactose by fermentation with *Torula lactosa*. With *Torulopsis candida* 50 grams of yeast containing 19 grams of protein were obtained from 100 grams of lactose.[10]

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# 4

# CONDENSED PRODUCTS

## INTRODUCTION

Skim milk, whey and buttermilk are condensed to reduce their shipping weight, to increase their usefulness in food manufacture and to preserve them. The plain condensed products are perishable but sugar or acid is often added to make modified concentrates which keep well. Products preserved by heat sterilization are discussed in Chapter 8. When milk fat, a stabilizer and certain other ingredients are added, such foods as condensed ice cream mix and concentrated milk spreads may be made.

Sweetened condensed milk has been produced throughout the world in various forms since Borden invented his process of evaporating milk under a vacuum in 1856.[17] The "Cajeta" of Mexico, the "Dulce de Leche"[96] of South America and the "Keer" of India (see Chapter 9) are concentrated by boiling the milk and sugar together in open kettles, often in very small batches. This heat treatment produces a food with a caramel flavor and color in contrast to the light color and delicate milk flavor of the domestic vacuum condensed product.

The natural fine flavor of milk is preserved better in the sweetened condensed milks, whey and ice cream mix than in the sterilized or dried products. Sterilization causes development of a cooked or caramel flavor, while drying makes the product susceptible to a type of staling not found when water is present. This fact should act as a stimulus to the development of more sweetened condensed foods.

**Concentration Procedures.** *Methods of concentrating* fluid milk products include evaporation of water, freezing and removal of ice and such fractionation methods as centrifugal separation and precipitation. Evaporation under vacuum is the only process that is widely used to manufacture condensed milk products.

*The vacuum condensing process* itself does not produce important changes in the milk. If skim milk is concentrated and then diluted with water to its original solids content it will show a small increase in acidity and refractive index and a slight decrease in freezing point.[51] The degree of dispersion of the milk protein appears to remain unchanged except under abnormal evaporation conditions. When plain skim milk is condensed to more than 45 percent solids, its viscosity increases rapidly, heat transfer in the vacuum pan is retarded, and there is a superheating effect which may denature the protein and result in rapid age thickening. Skim milk concentrated to 40 percent solids then reconstituted with water will show a slightly lower heat stability than the original milk.

A detailed description of different types of vacuum pans and their operation has been given by Hunziker,[64] and by Farrall.[38] The milk product boils in the evaporator at temperatures of 100 to 140°F corresponding to a range of gauge pressures between 28 and 24 inches of mercury. The vacuum pan is used because it protects the product from excessive temperature, it removes water rapidly and it is economical to operate. Vacuum pans are built in sizes capable of evaporating approximately 400 to 10,000 pounds of water per hour at costs of about 0.2 to less than 0.1 cent per pound of water.

The batch-type vacuum pan, like Borden's original retort, consists of a chamber in which the milk boils under vacuum and a jacket or coils through which heat is conducted to the milk. This design has been modified by the addition of inclined or vertical tubes in which the product is quickly heated before it is flashed into a vapor chamber for removal of the water vapor. Multiple-effect evaporators consisting of two, three or four units are now used in concentrating skim milk and whey. The multiple-stage evaporator is much more efficient than the single effect unit because each stage is heated by the exhaust vapor of the preceding unit and each unit is operated at a progressively lower pressure, the vacuum in the final stage being 27 to 29 inches. Vapor removed from the boiling milk first passes through an entrainment separator to recover traces of milk in the vapor. The vapor is then condensed in a wet or water spray condenser which requires a continuous supply of cold water.

A new low-temperature evaporator has been developed by Cross for use on citrus juices and other fluids.[29] Its value for the evaporation of milk products is now being determined. The liquid to be concentrated is heated by the latent heat of compressed ammonia vapor. The cooled ammonia is again vaporized to condense the water vapor removed from the liquid being concentrated. The ammonia vapor is then returned to the compressor to complete the cycle. The operations are conducted under very high vacuum and the liquid boils at about 60°F.

*Evaporation at atmospheric pressure.* Attempts have been made to devise small-scale, low-cost evaporating equipment that might be suitable for use on farms or in small milk plants. The atmospheric disc condenser developed during World War II is one example of such equipment.[9] Skim milk was reduced 75 percent in volume by placing it in a tank fitted with rapidly rotating discs half submerged in the milk. A stream of hot air was blown over the top half of the discs. The process may have been impractical, for the report was recalled shortly after it was issued.

An inexpensive small scale conical evaporator through which hot air is circulated and into which milk or whey is sprayed has been constructed and tested.[110] Equipment of this kind and a method for cooling and concentrating liquids, including milk and whey, has been described recently.[52] Small scale atmospheric concentrators designed like the large cone-shaped milk driers, but costing less than a vacuum pan, might provide small, isolated cheese factories with a means of whey disposal. However, any arrangement for concentrating small quantities of whey under atmospheric pressure would not be as efficient as vacuum evaporation.

The submerged combustion burner is being studied[110] to determine whether it can be used as an inexpensive means of concentrating small quantities of whey or skim milk. A mixture of air and natural gas is burned under the surface of the liquid. The heat of combustion causes evaporation of water from the whey or skim milk. Foaming may be reduced by the use of a foam depressant and by carrying out the evaporation in a wide-mouthed tank from which vapor can easily escape. The efficiency of water vapor removal is greater than when fuel is used to produce steam for heating. However, the quality of product concentrated under vacuum is much better than that condensed by an underwater burner.

*Concentration by freezing.* Fluid milk products may be concentrated by freezing part of their water content and removing the ice crystals. Whey has been concentrated to 30 percent solids, with a loss of 10 percent in the ice crystal sludge, by freezing it on a pilot plant scale.[2] Less energy is required to freeze water than to vaporize it. The latent heat of fusion of water is 143 B.T.U. per pound whereas its latent heat of vaporization is 971 B.T.U. But the cost of refrigeration and the mechanical difficulties attendant upon removing frozen ice crystals from milk have thus far retarded extensive commercial adaptation of systems of concentration by freezing. One process[4, 12] provides a large stainless steel cylinder surrounded by brine within which is mounted a screw conveyor. Scraping blades on the conveyor remove ice from the cylinder walls and carry it to a centrifuge that separates the ice from any milk that may have been carried over from the concentrator. By another process,[103] milk is frozen as a soft block which fits into the basket of a centrifuge. The concentrate is spun off, the ice is removed from the centrifuge and replaced by another block of frozen milk. The centrifugal separation of ice during the concentration of fruit juices by freezing has been described recently.[42]

Another method for the "freeze-concentration" of fluids provides for the controlled growth of ice crystals and their removal in such a way as to minimize contamination of the crystals by solids of the fluid being concentrated.[80]

Skim milk or whey could possibly be concentrated by exposing them to sub-freezing temperatures during suitable weather. This would provide small cheese factories with a cheap means of removing water from fluid whey. But uncertain weather conditions, the fact that heaviest whey production does not occur in the winter and the difficulties of removing the ice crystals render any natural freezing method of doubtful value.

*Concentration by centrifugal force.* Methods of concentrating certain milk ingredients by centrifugal separation have been devised. These are fractionation procedures and they are usually designed to concentrate only one constituent of the product. The fat, the casein complex, and denatured albumin may be removed by centrifugal force. Fat is concentrated in the ordinary cream separator. The casein which in milk exists as a calcium caseinate-calcium phosphate complex can be concentrated as a paste in high speed centrifuges that are now commercially available. Denatured whey protein can be centrifugally removed from whey on an industrial scale. Large quantities of water are used to help carry off the lactose of the whey while the protein fraction is gradually concentrated by means of a special centrifuge. The aqueous protein dispersion may be dried. Some processing details are presented in this chapter under Production Methods.

Ford and Ramsdell[40] have used the McBain air driven and air floated ultracentrifuge in studies of sedimentation velocities of milk components. By means of this tool these authors have characterized the colloidal particles as to size and composition. As a development of this work it may become possible in the future to precisely fractionate the colloidal or casein-containing proteins of milk and to isolate particles of certain sizes. Such fractionations may lead to the development of special products having specific characteristics as to body, texture, flavor or storage properties.

## TECHNICAL PROBLEMS

**Preservation of Condensed Products.** Plain condensed milk and sweet whey are perishable and must be held under refrigeration until they are used. These products are not suitable for long distance shipping or for storage of milk solids from season to season. After a preliminary heat treatment, it is customary to add either acid or sugar as a preserving agent to products which are to be kept for long periods of time. Such an addition confines the use of the milk concentrate to that class of foods with which the acid or sugar will blend.

*Heat* is the agent most widely used for preserving milk products. But when complete sterility is attained the heat treatment required is so severe that undesirable physical and chemical changes occur. (See discussion in Chapter 8.) The forewarming to which all the condensed products are subjected during manufacture helps to minimize the biological changes that might otherwise occur in storage.

The temperature to which a byproduct is forewarmed before it is drawn into the vacuum pan is generally 160 to 190°F; the holding time is 10 to 30 minutes. The heat treatment should be severe enough to destroy enzymes and to reduce the bacteria count. Care must be observed not to recontaminate the product after it is forewarmed, otherwise its keeping quality will be impaired. Hunziker[67] and Hammer[54] have discussed the types of bacteria and yeast that may cause spoilage in the condensed milks.

*Acid* is the active preserving agent in concentrated sour skim milk, buttermilk and whey and in such special products as concentrated spreads and salad dressings. The successive fermentations which occur in unheated milk are well known. Fresh milk at about pH 6.6 is first attacked by lactose fermenting bacteria which reduce the reaction to between pH 4.0 and 5.0. This reaction is unfavorable to bacteria but it does not inhibit the growth of yeasts and molds. Molds destroy the lactic acid and liberate alkaline products of protein decomposition which provide a favorable medium for the growth of putrefactive bacteria. The general procedure in the manufacture of products preserved by acid is to encourage the lactic fermentation in the medium, concentrate the mixture so that it contains approximately 5 percent lactic acid and then package it in air-tight containers to prevent mold growth.

It is important to know how much acid will inhibit bacterial growth. Data have been obtained as a result of studies to find the pH ranges favorable to growth of specific organisms and to determine the characteristic final pH attained by cultures.[7] Most organisms can tolerate media within the range pH 4.5 to 8.5 with values for optimum growth generally lying between pH 6 and 7. The final pH attained by bacterial cultures has been found by many investigators to vary with the composition of the medium. Rogers and Whittier,[99] for example, showed that the growth of *S. lactis* in milk was limited by both the concentration of hydrogen ions and the concentration of undissociated lactic acid. Many cultures do not reach a final hydrogen-ion concentration below pH 5.0 but in a favorable medium some organisms grow at reactions as low as pH 4.3 to 4.5. When acid is to be used as a preservative it may be assumed that undesirable growth will not occur in milk byproducts with a reaction below pH 4.5. However, hydrogen-ion concentration is only an inhibitory influence and it is not lethal except for very sensitive strains.

Products to be preserved by acid should always be pasteurized at some stage in their manufacture. When a product with a reaction of pH 4.5 is heated to 185°F for 10 minutes just before it is packaged in sterile containers, under sanitary conditions, it should not undergo bacteriological changes during storage.

*Sugar* acts as a preservative in sweetened condensed products by raising the osmotic pressure of their aqueous phase. A common formula for sweetened condensed skim milk is 30 percent skim milk solids which include 2.5 percent ash and 16 percent milk sugar together with 42 percent sucrose and 28 percent water. The sucrose in water concentration (the sugar ratio $= \frac{\text{sucrose}}{\text{sucrose} + \text{water}} \times 100$) of this mixture is 60 percent. This is the minimal sugar concentration that commercial experience has shown may be safely used. The milk salts and lactose help to increase osmotic pressure but only 4.7 percent lactose is soluble in the amount of water present in the above condensed skim milk.

Although much sweetened condensed milk is made with a sugar ratio of 60 percent it is generally considered that a sugar in water concentration of 62.5 percent will insure greater safety and superior keeping quality. Rice and Downs[94] considered that the sugar ratio should be between 63.5 percent and 64.5 percent. Hunziker[61] states that 62.5 percent is adequate to protect good quality milk from most bacterial deterioration but that a 64.5 percent sugar ratio is needed in products which are heavily contaminated with sucrose-fermenting yeasts. However, as Hunziker points out, proper forewarming and sanitary precautions are necessary if damaging fermentation during storage is to be avoided.

Corn sugar or glucose has been used as a preservative to replace part of the sucrose in sweetened condensed products. The osmotic pressure of a 10 percent glucose solution is approximately double that of a sucrose solution of the same concentration by weight. But Berkeley and Hartley[15] found that as the concentrations of the solutions of these sugars were increased, the osmotic pressure of the glucose solutions did not increase at the same rate as did the osmotic pressure of the sucrose solutions. Some of Berkeley and Hartley's data are plotted in Figure 5. Although the Boyle-Avogadro law requires the pressures of the solutions of both sugars to increase at a rate which is inversely proportional to the molecular weights of the dissolved substances, these curves show an even more rapid pressure rise than this. But it would be necessary for the glucose curve to reach a pressure of 140 atmospheres at 30 percent concentration in place of 48 percent to keep pace with the pressure rise of sucrose. It therefore follows that, if a 60 percent sucrose solution will exert sufficient osmotic pressure (140 atmospheres at 32°F) to inhibit bacterial growth in sweetened condensed milk, a concentration of 48 percent glucose will be required to produce an equal pressure. Recently Tarkow, Fellers and Levine[113] in a study of 30 to 50 percent sucrose and glucose solutions observed that in equal concentrations glucose was markedly more inhibitory than sucrose to test yeast and mold cultures. Mixtures of equal parts of the two sugars inhibited growth to a degree intermediate to that of either sugar alone at the same concentration. This is in agreement with the work of Ramsey, Tracy and Ruehe[91] who found that in concentrated solutions glucose was slightly more efficient than sucrose as a preserving agent.

Glucose is less soluble in water than sucrose, their respective solubilities at 60°F being 44 to 66.5 percent sugar solids. The amount of glucose which may be used in a sweetened condensed product is therefore limited by its low solubility.

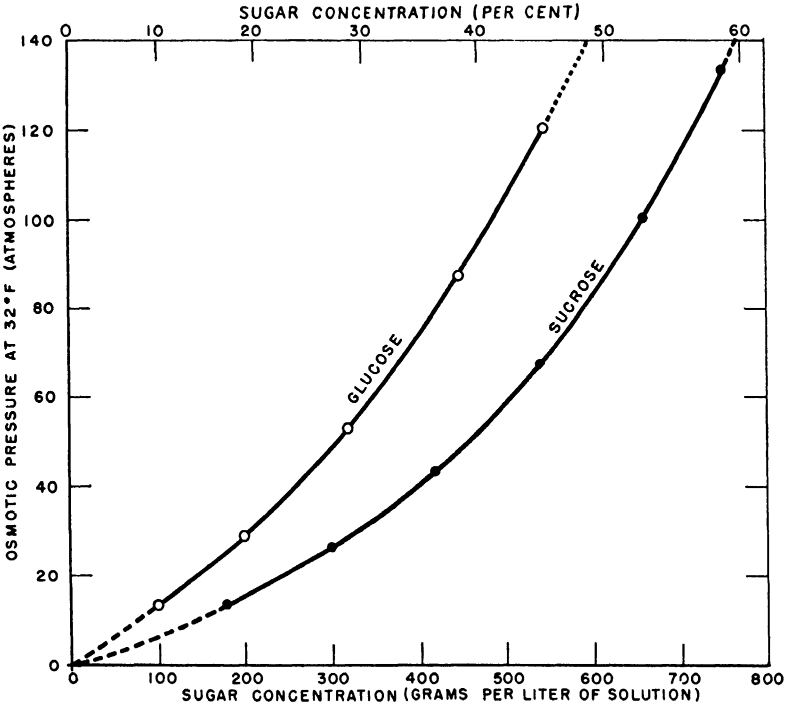

Fig. 5. The relationship between the osmotic pressure and the concentration of sucrose and glucose solutions. (*From data of Berkeley and Hartley.*[15])

A sweetened condensed skim milk which will keep bacteriologically can be made when half of the sucrose is replaced by glucose[91] but such a product will show much physical thickening during storage. If glucose must be used, it is advisable to replace not more than one-fourth of the sugar by the glucose.[61] The effect of glucose on physical thickening is discussed elsewhere in this chapter.

*Salt* was advocated by Williams[135] as a preservative for farm cream. Caulfield, Nelson and Martin[23] found 13 percent salt to be quite effective in retarding bacterial growth in cream over a ten-day storage period. Condensed whey may be preserved and held many months at room temperature if salt is added to the whey to produce a 15 percent salt concentration in the water of the whey.[122] However, the salt must be removed from these products before they can be used for most food purposes. This can be done with cream by the manufacture of plastic cream or butter. But until some way is found to utilize salty condensed products or to remove the salt, preservation by means of salt does not have practical value.

**Protein and Body Changes.** Important changes occur in the milk protein of condensed products during manufacture and storage and these affect their body or viscosity. Sweetened condensed skim milk should be viscous enough to retard the settling of lactose crystals but it should remain fluid enough during storage to pour and handle easily. The effectiveness of the superheating process as it is applied to plain condensed skim milk is judged by the viscosity which is developed in the product. Concentrated spreads must have a gel structure and this can be formed by the joint action of milk protein and a stabilizer. Other products are prepared so that they will have specific body characteristics.

*Nature of the thickening reaction.* The exact mechanism by which the viscosity of a milk protein suspension is increased or decreased is not understood. Thickening and the formation of a gel structure may be considered to correspond to the denaturation and agglutination of the protein. The denaturation reaction seems to be accompanied by either a mechanical or a chemical binding of water which thickens the fluid. Eilers et al.[35] found sweetened condensed skim milk to show "an almost purely viscous flow" when freshly prepared. The structure which developed during age thickening, however, was found to include a yield value. Sweetened condensed skim milk at this stage is thixotropic and can be temporarily thinned by mechanical agitation or homogenization. Whitaker and Hilker[133] found that when the viscosity of plain but superheated condensed skim milk was reduced by homogenization, the reduction did not affect the viscosity of ice cream mix made with the superheated condensed milk.

The heat thickening of condensed skim milk through the use of high temperatures and long forewarming time was shown by Greenbank et al.[47] to improve the baking quality of the concentrate when it was used in bread. When raw skim milk was forewarmed for 45 minutes at 149, 167, 185 and 203°F its relative viscosity after it was concentrated to 40 percent solids was 2.8, 4.5, 8.7 and 28 respectively.

When the thickening reaction proceeds to its maximal value the concentrated milk gel becomes insoluble and the protein will not redisperse in water to its original physical and chemical state. The rate at which these changes occur is controlled by the effects of temperature and concentration. The temperatures involved include those of forewarming, condensing, superheating, cooling and storage. The concentration of protein in the product during the different manufacturing operations affects its rate of denaturation. Eilers et al.[35] found that the viscosity of sweetened skim milk products increased so rapidly with increases in the solids content that even when the data were plotted with a logarithmic scale for the viscosity, a highly curved line was obtained.

The relationship between viscosity, concentration and the heat stability of milks is discussed in Chapter 8. The relationship between viscosity and agitation during heating is discussed in Chapter 5 and shown in Figure 8.

*Thickening by precipitation of casein.* The precipitated casein in soured skim milk or buttermilk will bind water, but to form a stable gel the milks must be partially condensed. This is done in the manufacture of concentrated sour skim milk and concentrated buttermilk. There is some difference in the character of the curd of these two products. Ordinary sour skim milk forms a hard curd which does not break up easily. However, the high acid concentration in the condensed skim milk has a solvent effect on the casein, softening the curd and giving the concentrate a smooth texture. Buttermilk curd, on the other hand, is naturally soft and the casein is in a finely divided state. Buttermilk can be readily concentrated to a smooth bodied product.

There is evidence that precipitation and redispersion of casein in some skim milk products may enhance their usefulness in manufactured foods. Baumann[13] devised a heat and chemical treatment for skim milk which made the protein appear to be more highly hydrated. This increase in water-holding capacity may have been accomplished by the conversion of the natural calcium caseinate of the milk into sodium caseinate. Skim milk was heated under pressure to 240°F for 10 minutes, then it was held at 210°F for 75 minutes. The reaction was adjusted to about pH 9 by addition of an alkaline sodium salt or sodium hydroxide. Finally the mixture was acidified to pH 6.5 to 8.5 and dried. This treatment increased the water absorbing properties of the finished product.

*Effect of forewarming on thickening.* The forewarming temperature to which the raw milk is first heated before it is concentrated for the manufacture of condensed milk affects the viscosity of the concentrate. Leighton and Deysher[70] found that while forewarming temperatures of 200 to 210°F stabilized evaporated milk toward heat, sweetened condensed skim milk was most stalbe at about 150°F and age thickening increased as the forewarming temperature was raised to 210°F. From this and more recent work[26, 47, 65, 109, 126] certain generalizations may be drawn.

Milk heated to 145°F before condensing produces a thinner, plain or sweetened condensed milk than when other temperatures up to boiling are used. The viscosity of plain and sweetened condensed milk and the age thickening of the sweetened milk increase progressively as the temperature of forewarming is raised to 212°F. Temperatures above boiling again decrease the viscosity of the concentrated milk. Quantitative data on the time-temperature-viscosity relationship at temperatures from 212 to 300°F have not been published. However, the data of Table 5 give some indication of the stability effect of high temperature forewarming. Since temperatures of forewarming below 175°F should not be used because of the survival of certain bacteria, yeasts and molds, milk to be made into plain or sweetened condensed milk is generally heated to 175 to 185°F before it is condensed. It is of interest to note that the forewarming treatment has the same effect on the viscosities of both plain and sweetened condensed milk.

TABLE 5. *Effect of High-temperature, Short-time Forewarming on the Viscosity of a Sweetened Condensed Skim Milk Held in Storage at 60°F* (Sugar ratio approximately 60)[126]

| Sample No. | Forewarming Conditions | Total Solids, Percent | When stored for— | | | |
|---|---|---|---|---|---|---|
| | | | 1 day (Poises) | 24 days (Poises) | 58 days (Poises) | 124 days (Poises) |
| 1 | 180° for 10 minutes | 71.44 | 360 | 534 | 853 | >1,100 |
| 2 | 180° for 10 minutes followed by 240° for 30 seconds | 72.78 | 220 | 275 | 369 | 534 |
| 3 | 180° for 30 minutes followed by 240° for 30 seconds | 71.54 | 50 | 116 | 182 | 457 |
| 4 | 240° for 30 seconds | 70.19 | 20 | 30 | 69 | 264 |

Forewarming temperatures above 212°F have been utilized to advantage in the manufacture of sweetened condensed milk when it was to be subjected to long storage at temperatures above 75°F. It has also been suggested on the basis of unpublished data[124] that less sugar and more milk solids might be used in sweetened condensed products if the raw milk was forewarmed at temperatures above 212°F.

*Thickening by superheating.* Superheated condensed skim milk is sometimes used in ice cream manufacture where it is said to increase the viscosity of the mix and give the finished ice cream a more resistant body and a smoother texture.[35] Its popularity for this purpose has decreased in recent years. Condensed skim milk is superheated by subjecting it to temperatures near boiling until the casein approaches the first stage of coagulation and the milk becomes viscous and even gel-like. For most purposes the superheating is stopped as soon as the viscosity is doubled or tripled and before gelation sets in. The factors which affect the thickening of condensed milk during superheating are essentially those which control its heat stability. (See Chapter 8, Figure 14). High concentrations of solids and high acidity cause the milk to thicken faster and at a lower temperature and in a shorter time than when the milk is of low concentration and acidity. The quantity of heat to which a milk is subjected during superheating should be varied according to the condition of the milk and the viscosity that is required.

Mook[78] has described a process for superheating condensed milk products at temperatures of 170 to 300°F until the protein is actually coagulated and slightly toughened. The coagulum is "smoothed out" and the dried and reconstituted product has a viscosity 4 to 8 times that of the untreated milk. The process appears to take full advantage of the water-holding capacity of casein which has been heat coagulated at a concentration of 35 to 45 percent milk-solids-not-fat. If this process were carried too far, whey would soon be expressed from the casein, the viscosity would decrease and the curd would become firm and tough.

*Thickening on aging.* Age thickening of sweetened condensed whole or skim milk proceeds until a gel structure develops. The time required for the milk to change from a fluid to a gel state depends on the quality of the raw milk, the manufacturing processes employed, the composition of the finished milk and the conditions of storage to which the product is subjected. When sweetened condensed milk age thickens to a viscosity of 600 to 800 poises, it is generally no longer suitable for use in high-grade food products. Off flavors often develop and the milk is too viscous to handle easily. A soft gel structure is present at 1000 poises.

The viscosity of sweetened condensed milk has been found to increase logarithmically with increases in storage temperature and arithmetically with increases in storage time.[129] For conditions of constant viscosity, time varied logarithmically with temperature. Some of these relationships are shown in Figure 6. It has been suggested that viscosity values could be predicted by applying these relationships to time-temperature data from high temperature, accelerated storage tests.

The viscosity of sweetened condensed milk held under the same conditions of storage increases a little more rapidly in milks packed in small cans than in milks packed in barrels.[129]

*Effect of sugars on thickening.* Sugars react with milk protein to affect the body and color of the condensed milks. Although it has been shown[88] that small changes in viscosity, surface tension, heat stability and buffer value occur as a result of the addition of sugar to milk, there is no information as to the specific chemical effect that the sugar may have upon the protein. Leighton and Leviton[71] have pointed out that the addition of a highly soluble non-electrolyte such as sucrose to the colloidal suspension, skim milk, may so increase the volume of the liquid phase and dilute the suspended protein that the actual viscosity of the whole system will be lowered. This principle was applied by Leighton and Leviton to the separation of milk sugar from highly concentrated skim milk. The thinning effect of sugar can also be demonstrated by making two batches of sweetened condensed milk using 16 pounds and 18 pounds of sugar, respectively, per 100 pounds of fresh milk. When such mixtures are condensed to the same milk solids content, the milk having the less sugar will have the higher viscosity. Hunziker[59] found that the tendency for unstable milk to age thicken could be decreased by increasing its sugar content.

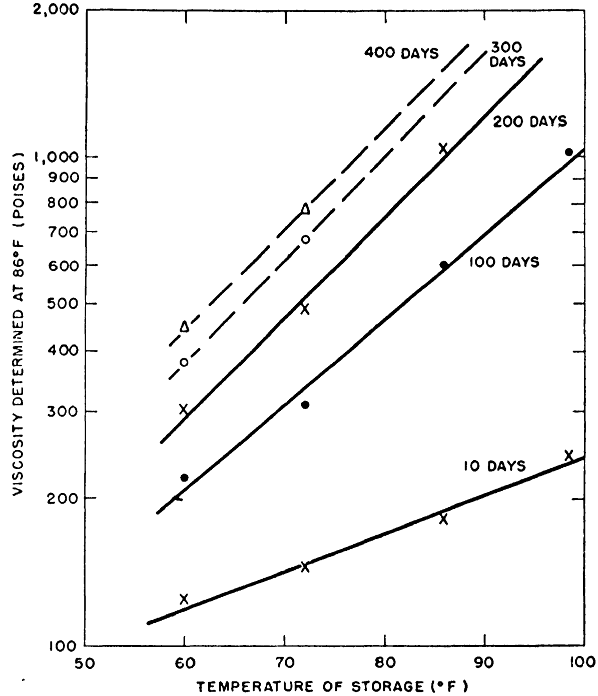

Fig. 6. The effect of time and temperature of storage on the viscosity of sweetened condensed milk.[129] The samples were part of a large commercial batch and contained 9.61% fat, 23.91% milk-solids-not-fat and 41.65% sugar. They were packed and held in 2-ounce cans. Viscosity determinations were made in a uniform manner by means of a McMichael viscosimeter.

Glucose, corn sirup and allied products, if used in the sweetened condensed milks, cause more rapid age thickening than does sucrose. Condensed milks containing from 25 to 50 percent substitution of corn sweeteners for sucrose become excessively thick and dark in color during storage at 70°F for 2 months or longer. Such milks remain in a satisfactory condition only if they are held in storage at temperatures below 45°F. A sweetened condensed milk of acceptable body and color may be made by replacing up to 25 percent of its sucrose with corn sugar. This product should be stored below 60°F for not longer than 2 or 3 months. A sweetened condensed milk made entirely with sucrose will have a better flavor as well as color and body after storage at any temperature above 40°F than a milk made with corn sugar. Sucrose is still the best preserving ingredient that has been found for the manufacture of concentrated milk. The corn products have been used largely because they have been readily available and cheaper than sucrose. In some food products corn sweeteners are used for the desirable effects they have on the body of the food. Ice cream is an example of one of these.[27]

The time of adding either sucrose or corn sweeteners to the batch has an effect on the thickening of sweetened condensed milks. The greatest thickening tendency results when the sugars are added to the milks before forewarming. There is less effect if the addition is made after forewarming as the last milk is drawn into the pan. For best results the sugars should be made into a water sirup, heated to boiling and drawn into the pan after the milk.

*The color* of the sweetened condensed products darkens with age. The reaction responsible for this darkening is the same as that which causes the browning of other milk products; it is a reaction between the sugars and the amino groups of the proteins. A detailed discussion of the browning reaction is presented in Chapter 8. Sweetened condensed milk seldom becomes unfit for use simply because of a darkening in color. But the dark color generally is an indicator telling of improper heating or storage conditions that result in thickening and gelation.

Acidified concentrated milks do not darken in color as do the sweetened condensed milks. The sugar-amino-acid-condensation reaction proceeds very slowly as the reaction of the milk drops below pH 5. Concentrated sour skim milk and condensed buttermilk have an acidity of about 5 percent and a reaction below pH 4.6. They do not become brown during manufacture or during storage.

*Freezing as a cause of protein change.* During prolonged storage frozen condensed skim milk undergoes an important physical change that is caused by gradual denaturation of the milk protein. The conditions which cause the changes in the protein have been investigated[3, 10, 11, 28, 33, 34, 81, 92, 93, 125] but the mechanism of the change is still obscure. The effect of the denaturation is to cause the protein to become insoluble and incapable of redispersing normally in the milk when it is thawed. The presence of denatured protein is noticeable sooner in the condensed than in the plain skim milks.

The approximate time of the appearance of protein denaturation in some plain and condensed skim milks is shown in Table 6. A gel structure forms in skim milks condensed to one third or less of their original volume after all the casein becomes insoluble. The insoluble protein may be redispersed by homogenization but, if the milk has been in storage at 0°F for six months or longer, the casein will not remain permanently dispersed. Ice cream that contains protein in this condition shows a curdy appearance in melt-down tests.

The temperatures of storage affect the rate at which protein denaturation occurs in frozen milk. The lower the temperature of storage the longer the protein denaturation is delayed. Anderson and Pierce[3] found 17 grams of wet precipitate in raw skim milk held frozen for 6 months at 10°F. Milk held under the same conditions at −14°F showed only one-half gram of precipitate. Condensed skim milk may be held at −20°F three or four times as long as 0°F before detrimental changes become noticeable. The temperature of storage should be held constant and not allowed to fluctuate.

TABLE 6. *Approximate Time of Protein Denaturation during Storage of Frozen Condensed Skim Milk at 0°F*[1]

| Solids Content of Skim Milk, % | Time of storage at 0°F | |
|---|---|---|
| | Time until Precipitate Appears (weeks) | Time until a Curdy Structure Develops[2] (weeks) |
| 9 | 12 | 37 |
| 18 | 6½ | 18 |
| 27 | 3 | 9 |
| 36 | 1½ | 5 |

[1] The data in this table are based on results given in the papers cited in the accompanying discussion on frozen skim milk.

[2] Quantitative data are not available. The figures refer to the time required to denature most of the casein. The thawed milk presents a curdy mass which may sometimes be redispersed by homogenization.

**Lactose Crystallization.** Lactose crystallizes from solution in many of the byproducts which are prepared in concentrated form. When the solubility of lactose in the water of a product is exceeded in the preparation of the concentrate, it is customary to force the lactose to crystallize quickly in order to produce small crystals and thus avoid sandiness. To do this the concentrate is cooled quickly to obtain a supersaturated sugar solution, then it is seeded with pulverized lactose crystals and stirred until an innumerable number of small crystals have formed.

Although lactose has a low solubility it is capable of forming highly supersaturated solutions in water and concentrated milk products. The conditions of temperature and concentration best suited to the production of small crystals have been approximately determined by commercial experience and by research. Additional investigations on the existence and location of a definite supersolubility curve and on the effect of sucrose and other milk constituents on lactose solubility are needed.

Leighton and Peter[73] determined a supersolubility curve for lactose and found it to be in the neighborhood of 54°F below the final solubility curve. Following Ostwald's definitions this gives a metastable zone between the final and supersolubility curves and a labile zone beyond the supersolubility limit. In the metastable zone seeding is required before crystallization can take place, whereas in the labile area spontaneous crystallization will occur. It is not clear how much real difference exists between these two zones. Herrington,[56] after an extensive study of lactose crystallization concluded there was no sharp dividing line between the metastable and labile zones in supersaturated lactose solutions.

Peter[85] showed that the solubility of lactose was decreased in the presence of sucrose. This occurs in products such as ice cream and sweetened condensed milk. Peter worked at 26.6 and 32°F and found the solubility of lactose in saturated sucrose solutions to be decreased to about one-half of its solubility value in water. However Hunziker and Nissen,[68] working at 50 and 65°F, temperatures more common to sweetened condensed milk, found the solubility of lactose in 62 percent sucrose solutions to be 15 percent less than in water.

In view of the state of our knowledge on the supersolubility of lactose and on its solubility in the presence of sucrose, salt solutions,[57] and milk colloids[68] it is difficult to definitely fix a crystallization procedure for sweetened condensed products. However, a few figures calculated from available data will be of interest.

The calculated concentration of lactose in water which would exist in some concentrated products if the lactose were completely dissolved is shown in Table 7. When these values are compared with the solubility of lactose given in Table 8, the approximate degree of supersaturation of each product at various temperatures can be determined. The final and supersolubility values of Table 8 were calculated from data of Hunziker and Nissen which indicate that the solubility of lactose is 15 percent less in 62 percent sucrose solution than in water. The condensed wheys reach the supersolubility zone at vacuum pan temperature, 120° to 140°F. Sweetened condensed skim milk enters the supersolubility region when it is cooled below 102°F. The lactose in plain condensed skim milk with a solids content below 30 percent will not crystallize even at 32°F unless the milk is seeded with lactose. Skim milk of 42 percent solids such as is used for spray drying will not enter the labile crystallization area unless it is cooled to about 60°F, or lower.

The optimum temperature to which a condensed product should be cooled before it is seeded and held with stirring to crystallize the lactose is that temperature at which it enters the supersolubility zone. Hunziker[62] has drawn a forced crystallization curve for sweetened condensed milk which yields values near those presented in Table 8 for the supersolubility of lactose in 62 percent sucrose solutions. Whittier and Gould,[134] as a result of their studies on lactose solubility, outlined a simple procedure for lactose crystallization. They found that the rate of separation of lactose from its supersaturated solutions during the first 2½ hours was greater at 86°F

TABLE 7. *The Calculated Lactose-in-water Concentration of Some Concentrated Byproducts in which the Lactose is Completely Dissolved.*[58,68,73]

| Product | Composition of Byproduct | | | | Lactose in water,[1] % |
|---|---|---|---|---|---|
| | S.N.F., % | Lactose, % | Sucrose, % | Water, % | |
| Sweetened condensed skim milk | 30 | 16.1 | 42 | 28 | 36.5 |
| Sweetened condensed whey | 38 | 28.5 | 38 | 24 | 54.3 |
| Plain condensed skim milk | 27 | 14.5 | | 73 | 16.6 |
| " | 30 | 16.1 | | 70 | 18.7 |
| " | 36 | 19.3 | | 64 | 23.2 |
| " | 42 | 22.5 | | 58 | 27.9 |
| Plain condensed whey | 65 | 48.6 | | 35 | 58.1 |

[1] Lactose in water $= \dfrac{\%\ \text{lactose}}{\%\ \text{lactose} + \%\ \text{water}} \times 100.$

TABLE 8. *The Solubility of Lactose in Water and in 62 Percent Sucrose Solutions*

| Temperature | | Lactose Solubility | | | |
|---|---|---|---|---|---|
| | | In pure Water | | In water containing 62% Sucrose | |
| °C | °F | Final[1] % | Supersolubility[2] % | Final[3] % | Supersolubility[3] % |
| 0 | 32 | 10.6 | 19.9 | | |
| 10 | 50 | 13.1 | 24.6 | 11.1 | 20.9 |
| 20 | 68 | 16.1 | 30.4 | 13.7 | 25.8 |
| 25 | 77 | 17.8 | 33.7 | 15.1 | 28.6 |
| 30 | 86 | 19.9 | 37.0 | 16.9 | 31.4 |
| 40 | 104 | 24.6 | 43.9 | 20.9 | 37.3 |
| 50 | 122 | 30.4 | 51.0 | 25.8 | 43.4 |
| 60 | 140 | 37.0 | 59.0 | 31.4 | 50.2 |

[1] Data from Hudson.[58]

[2] Calculated from data of Leighton and Peter.[73]

[3] Calculated from solubilities in water based on data of Hunziker and Nissen.[68]

than at 77°F or any lower temperature. After seven hours the amount of lactose which separated at 77°F approached that which separated at 86°F. If the milk is cooled to 86°F, seeded and stirred at this temperature for at least an hour the crystal structure will generally be satisfactory.

The success of the modern method of pumping sweetened condensed milk from the pan through cooling coils to the final containers seems to be dependent upon the rapid formation of many crystal nuclei in the cooling coils. The milk is cooled from between 110 and 130°F to between 50 and 60°F in the coils in about 10 minutes. Lactose crystals in the coils seed the milk as it becomes highly supersaturated during the cooling process. The tiny crystals which form in the coils are scarcely visible under the microscope. As the crystallization process proceeds in the final container at 50 or 60°F, lactose deposits are made on the original nuclei formed in the cooling coils. These remain small and do not grow to objectionable size provided a large number of the nuclei were originally formed.

TABLE 9. *Relationship between the Number and Size of Lactose Crystals and the Degree of Sandiness of Sweetened Condensed Milk*[1]

(Measurements and calculations by Sparks[108])

| Crystals per Cubic Millimeter, Number | Calculated Length of Longest Edge of Crystal, Microns | Scale of Sandiness |
|---|---|---|
| 400,000 | 9.3 | Excellent |
| 300,000 | 10.25 | Good |
| 200,000 | 11.7 | Slightly pasty |
| 100,000 | 14.75 | Fine mealy |
| 50,000 | 18.6 | Mealy |
| 12,500 | 29.4 | Sandy |

[1] Data and calculations from milk containing 9% fat, 22.5% S.N.F. including 12.2% lactose, 42.5% sucrose, 26% water

Sparks[108] counted the number of crystals of lactose in milks of various degrees of sandiness and calculated the crystal size in each milk. Some of his results are reproduced in Table 9. If a milk is to have a smooth body the crystals should be below 11 microns in size.

## PRODUCTION METHODS

**Skim Milk Concentrates.** The skim milk concentrates including the important products, plain and sweetened condensed skim milk, utilize less than 5 percent of the annual national production of skim milk. Nevertheless, they are byproducts which are very popular in food manufacture.

*Plain and superheated condensed skim milk.* Skim milk is concentrated and sold in bulk though perishable form for use in food manufacture. It is an excellent source of non-fat milk solids in ice cream, bakery goods and confections. It may be used to standardize evaporated milk and it is an intermediate form in the manufacture of dry skim milk. For these uses skim milk is generally condensed to concentrations between 20 and 40 percent solids content.

*Manufacture* of these products is carried out by a well-known and simple procedure. The milk is forewarmed, condensed and then, if desired, superheated. When the product is to be used in ice cream or in other foods where a cooked flavor is not wanted, the forewarming should be carried out at 160 to 170°F for 10 minutes. Skim milk which is to go into bakery goods and confections should be forewarmed at 180 to 190°F for 10 to 20 minutes. When the condensed skim milk is to be superheated, a forewarming temperature of about 150°F held for 20 minutes is generally sufficient.

TABLE 10. *Density and Weight per Gallon of Plain Condensed Skim Milk*

| Total Solids, % | Density of Skim Milk | | | | Weight per gal. at 50°F, Pounds |
|---|---|---|---|---|---|
| | 120°F | | 50°F | | |
| | °Baumé | Sp. gr. | °Baumé | Sp. gr. | |
| 20 | 9.1 | 1.0670 | 11.0 | 1.0821 | 9.02 |
| 22 | 10.1 | 1.0749 | 12.0 | 1.0902 | 9.09 |
| 24 | 11.2 | 1.0837 | 13.1 | 1.0993 | 9.16 |
| 26 | 12.2 | 1.0919 | 14.1 | 1.1077 | 9.23 |
| 28 | 13.3 | 1.1010 | 15.2 | 1.1171 | 9.31 |
| 30 | 14.3 | 1.1094 | 16.2 | 1.1258 | 9.39 |
| 32 | 15.4 | 1.1188 | 17.3 | 1.1355 | 9.47 |
| 34 | 16.4 | 1.1275 | 18.3 | 1.1444 | 9.54 |
| 36 | 17.5 | 1.1373 | 19.4 | 1.1545 | 9.62 |
| 38 | 18.5 | 1.1462 | 20.4 | 1.1637 | 9.70 |
| 40 | 19.6 | 1.1563 | 21.5 | 1.1741 | 9.79 |

The hot milk is drawn into the vacuum pan and condensed in the usual way. The density of plain condensed skim milk between 20 and 40 percent solids content in given in Table 10. The pan temperature is approximately 120°F but a correction of 0.027 to 0.03° Baumé for each degree Fahrenheit may be used when the temperature deviates from 120°F. The density and weight per gallon on condensed skim milk are also given at 50°F, a common temperature for storage and handling of this product.

Condensed milk may be superheated in an open vat equipped with a means of heating and agitating, in a tilting or candy kettle having a fast double action scraper and stirrer, or in a vacuum pan. Heating is generally done by injecting cleaned, live steam directly into the milk, but the slower method of heat transfer through coils or jackets may also be used. To superheat in the vacuum pan at the end of the condensing operation, the vacuum should be broken, the milk heated and then quickly cooled by again evacuating the pan. The rapid removal of water vapor from the hot milk cools it at once to about 120°F. The cooling operation should be finished in a properly designed coil vat or other container with a strong agitator.

The process of superheating condensed skim milk has often been described.[36, 63, 107, 116] One procedure which might be used to manufacture superheated condensed skim milk will be given, but modifications to suit special conditions may be necessary. Forewarm the skim milk to 150°F for 20 minutes, concentrate it to 32 percent solids content, cut off the steam, water and vacuum to the pan and turn the steam into the milk through the superheating valve. Heat the milk to 190°F and hold for 10 minutes. Start the vacuum pump and remove vapor until the temperature drops to 120°F. Draw out the milk and finish cooling it in a coil vat. The finished product should be very viscous but not coagulated. It should test about 30 percent total solids content.

A process for superheating milk for drying and subsequent use in ice cream has been patented by Mook.[78] The milk is forewarmed at about 170°F, condensed to 35 to 45 percent solids-not-fat and then superheated within the range 180 to 210°F by direct steam, or in a jacketed kettle. Superheating continues until a light, slightly toughened coagulum forms, after which the product is dried.

*Sweetened condensed skim milk* is a standard commercial product.[66] It is one of the best forms of skim milk solids for shipping and storage. The skim milk is forewarmed to 175 to 190°F for about 10 minutes and then drawn into the vacuum pan. The sugar, preferably prepared in the form of a filtered sirup which has been boiled with an equal weight of water, is drawn into the pan after the milk. The dry sugar may also be added to the last hot milk which is drawn into the pan. Enough sugar should be used to give a concentration of at least 60 percent sucrose in the water of the concentrate. Milks of various compositions may be produced but a common commercial product contains 42 percent sugar and 30 percent skim milk solids. If the raw skim milk contains 9 percent solids, 12.6 lbs. of sugar per 100 lbs. of skim milk will be required. This is the minimal quantity of sugar which can be safely used. Thirteen pounds of sugar per 100 lbs. of skim milk will lessen age thickening and safeguard the product against bacterial spoilage. The milk is condensed to 72 to 74 percent solids content at a density of 35.7 to 38°Bé.

Cooling and crystallization of the lactose is generally accomplished by either a batch or continuous type cooler. In the batch system, the milk is agitated in jacketed cans, vats, or tanks while cold water is circulated around the walls of the container. Some vats are equipped with a helical coil through which a cooling liquid may be circulated. The milk should be cooled as rapidly as possible to 90°F, seeded with finely ground lactose crystals, then held between 86 to 90°F with slow stirring for one hour. Cooling is then continued until a temperature below 60°F is reached, when the milk is ready to be placed in cans or barrels. The storage temperature should be below 60°F.

Continuous cooling of sweetened condensed skim milk is done by pumping the milk from the vacuum pan or drop tank through a submerged coil or internal tubular, counter-current cooler. The milk is seeded from the film of milk of the previous day's make which coats the cooler coils. If the coils are clean, they may be coated with seed material by pumping a few gallons of finished milk through them ahead of the new batch. In the continuous method of cooling, the milk is cooled to 60°F in about 8 to 12 minutes and filled into bulk containers at once. Spontaneous crystallization is started in the cooling coils and the crystallization becomes complete during the first 24-hour storage period.

Vacuum cooling by either the batch or continuous method has recently been developed.[60, 74] The milk is drawn from the vacuum pan into another vacuum chamber which is equipped with an agitator. The vacuum is increased from normal to about 28.5 inches of mercury corresponding to a temperature of 89.5°F. The batch is seeded and agitated to promote crystallization of the lactose, then cooling is continued until a temperature below 70°F is reached (about 29.2 inches of mercury). The milk should finally be cooled to 60°F, or lower, before it is stored. Vacuum cooling, while showing unusual promise, is still in the development stage. It does not have the background of years of proven commercial usage that have made the conventional methods universally acceptable.

*Frozen condensed skim milk* is plain condensed skim milk that has been frozen and held in frozen storage. It is used largely in ice cream manufacture. The skim milk should be forewarmed at 160°F for 15 minutes, then condensed to 30 percent solids, cooled and run into cans suitable for freezing. For easy handling and rapid freezing and thawing the containers should not be larger than 5-gallon capacity. Freezing should be done at as low a temperature as possible, but not above 10°F, and preferably below −10°F. The frozen condensed skim milk should be held in storage at the temperature at which it is frozen. The storage temperature should not fluctuate.[10] High temperatures of storage and variations in the storage temperature accelerate the rate at which the protein becomes insoluble. The storage time that will produce insoluble protein varies from about 3 weeks at 10°F, to 3 or 4 months at −10°F, and may be even longer at lower temperatures.

*Low-lactose or milk protein concentrates* are skim milk derivatives from which part or all of the milk sugar has been removed. They were developed to enable the ice cream manufacturer to improve the body and texture of his ice cream by increasing the milk protein content of his mixes. If ice cream is made to contain more than 10½ or 11 percent milk-solids-not-fat (about 5.8 percent lactose), sandiness or lactose crystallization may occur during storage. But by the use of a low-lactose product the milk protein content of a mix may be raised to the equivalent of more than 12 percent milk-solids-not-fat without production of sandiness. Another method of avoiding sandiness has been suggested by Gray and Turnbow[45, 46, 119] which consists of adding dry skim milk to the mix during freezing. The preparation of ion-exchange treated skim milk for use in ice cream is discussed in Chapter 9. Both the soluble and coagulated whey protein products mentioned later in this chapter could be adapted to ice cream manufacture.

Processes for the preparation of low-lactose or milk protein concentrates from skim milk may be divided into three groups: (a) enzymatic hydrolysis of the lactose; (b) removal of lactose from the milk; and (c) separation of the casein from the milk and its redispersion in water, milk or ice cream mix. Method (c) eliminates the milk salts and the water-soluble albumin together with the lactose but the method usually involves partial or complete denaturation of the casein.

(a) *Enzyme hydrolysis* of the lactose in skim milk by the enzyme lactase is an excellent method of preparing a low-lactose concentrate. The resulting products, glucose and galactose are sweeter and more soluble than lactose. Such a low-lactose concentrate was suggested for use in ice cream by Turnbow.[120] However, lactase has not been available at a reasonable price and in commercial quantities.

One careful preliminary investigation[19] failed to obtain active lactase preparations which were suitable for industrial purposes. Almonds, snails, cockroaches, animal intestines and preparations of yeasts, molds and bacteria were examined for lactase. The best results were obtained with a culture of *T. cremoris* (American Type Culture Collection #2512). The organism was grown at about 86°F in sterile, clarified whey (pH 4.5), fortified with small quantities of ammonium sulfate and ammonium primary phosphate. The yeast cells were separated by collecting them in the bowl of a milk clarifier. The cells were subjected to mechanical disintegration by shaking in sand according to the method of Curran and Evans[30] and a clear extract was prepared by centrifuging the cell mixture. This extract, which did not always contain the active enzyme, was tested for its ability to hydrolyze lactose. It was concluded that lactase was not as efficient in hydrolyzing lactose as invertase was in hydrolyzing sucrose; that the optimum reaction for the hydrolysis was about pH 5; that lactase was extremely unstable when it was separated from the cell and that the enzyme was not present in the materials examined in commercially significant quantities.

Recently, lactase prepared by Rohm and Haas[100] has been made available for pilot plant tests. In its present form a quantity of enzyme equal to from 0.5 to 3 percent of the weight of lactose in the skim milk or whey

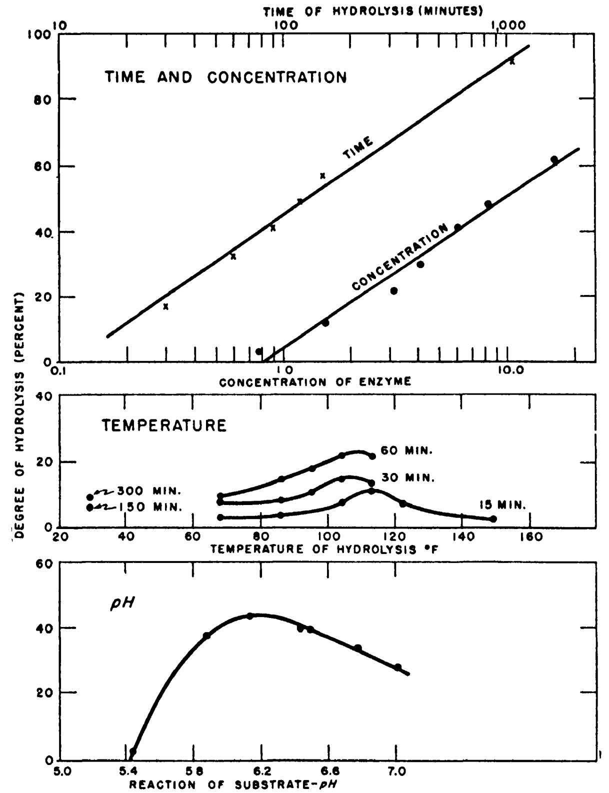

Fig. 7. Activity curves for the hydrolysis of 10 percent lactose solutions by lactase. Plotted from data furnished by Rohm and Haas.[100] The "lactase A" was prepared by Rohm and Haas from yeast. The quantity of enzyme preparation used in each experiment, expressed as a percentage of the weight of lactose in the substrate, was: time curve, 4.0%; temperature curves, 3.1%; and pH curve, 6.5%. The time curve was determined at 104°F, and pH 6.5. The concentration of the enzyme used to determine the concentration curve is expressed as grams of enzyme per 100 grams of lactose.

being treated is required to hydrolyze about half of the lactose in the product. The rate and extent of hydrolysis depends upon the operating conditions. Characteristics of the enzyme lactase are shown in Fig. 7. The effectiveness with which the Rohm and Haas preparation will hydrolyze the lactose in skim milk is shown in Table 11.

(b) *Removal of lactose* from the milk without denaturing the casein produces a product which is well adapted to ice cream and food uses.[71, 72, 130] The addition of cane sugar in suitable amounts to skim milk lowers the viscosity of the concentrated product. This may be attributed to the diluting action of the sugar since it increases the volume of the liquid phase of the suspension. The lowering of viscosity facilitates lactose crystallization and aids in removal of the crystals. The optimum quantity of cane sugar to add to 100 pounds of skim milk for maintenance of minimal viscosity is 5.9 pounds.

To manufacture low-lactose milk, the mixture of skim milk and sugar should first be forewarmed to 150°F for 10 minutes. It is then drawn into the vacuum pan, where a concentration of 68 to 70 percent total solids is attained. A striking cup may be used as an aid in striking the batch. The specific gravity of the finished milk at 104°F should be between 1.32 and 1.34, and its viscosity at this temperature will range between 26 and 32 centipoises. The refractive index of the condensed product at 104°F should be approximately 1.47 to 1.49. After it is drawn from the pan, the concentrated milk is next cooled to between 68 and 77°F in cold water with occasional slow stirring. It is then held about 20 hours in a refrigerator at 50°F; this temperature being preferred to a higher temperature (where maximum crystallization would occur) in order to prevent the development of excessive viscosity. During this time the lactose crystallizes and can be removed from the milk by means of a sugar centrifuge.

TABLE 11. *Hydrolysis of Lactose in Skim Milk of 30 percent Solids Content with a Lactase Preparation*

(Enzyme and data from Rohm and Haas Co.)[100]

| Concentration of Enzyme Preparation, % of lactose in skim milk | Temperature of hydrolysis, °F | Time of hydrolysis, hr. | Time of hydrolysis, days | Degree of hydrolysis, % |
|---|---|---|---|---|
| 3 | 104 | 1 | | 14.5 |
| 3 | 104 | 2 | | 22.8 |
| 3 | 104 | 4 | | 46.0 |
| 3 | 40 | | 1 | 18.5 |
| 3 | 40 | | 4 | 47.0 |
| 3 | 40 | | 5 | 51.0 |
| 0.6 | 40 | | 1 | 2.7 |
| 0.6 | 40 | | 4 | 15.0 |
| 0.6 | 40 | | 5 | 21.0 |
| 0.6 | 40 | | 11 | 41.0 |

The separation of the lactose crystals is the only step in the process which offers any serious obstacle to the preparation of low-lactose milk by any dairy plant equipped with a vacuum pan. Special equipment must be used to remove the small lactose crystals from the heavy condensed milk. When the viscosity of the product is not excessively great, filter pressing is a rapid means of separating the lactose. But for all viscosity conditions and crystal sizes, centrifugal separation is preferable. The filter cloths should be about 200 mesh for either centrifuge or filter press.

The quantity of lactose which can be removed from the condensed milk will vary from 40 to 75 percent of the total lactose present in the milk. Under favorable conditions an average of 60 percent may be expected.

There is less tendency toward bacterial spoilage in low-lactose milk than in plain condensed skim milk because of the added cane sugar. The sugar ratio will vary in low-lactose milk from 45 to 50 which is considerably below the required figure of 63 necessary to prevent bacterial growth. Additional cane sugar may be added to preserve the milk or the product may be dried.

The viscosity of low-lactose milk increases during storage, a high viscosity developing more rapidly where the temperature of storage is high. For routine production the finished product may be diluted with plain skim milk to attain a uniform concentration of about 50 percent total solids. By thinning the milk in this manner the inconvenience of handling a heavy bodied product is avoided.

Thorneloe[115] has modified this process to facilitate crystal growth and separation. The skim milk-sugar mixture is concentrated to about 72 percent solids, dropped into a crystallizing tank and held about 20 hours at room temperature without cooling. The lactose crystallizes spontaneously without seeding or stirring and the crystals grow to at least 100 microns in size. The viscous milk is then diluted and thinned with a sucrose sirup containing about 66 percent sugar and centrifuged quickly to remove the lactose crystals. The approximate composition of the finished low-lactose milk is sugar 40.8 percent, lactose 9.2 percent, protein and salts 16.5 percent and water 33.5 percent.

A somewhat different approach to the problem of obtaining a low-lactose product for use in ice cream has been devised.[123, 125] Cream of 25 percent fat is homogenized at 150°F and 2500 pounds pressure, cooled and frozen. The frozen product is then thawed at 50°F, the clear serum being drained away. The mass of fat and protein which remains contains approximately 65 percent fat, 6 percent protein, 1 percent lactose plus ash and water. The protein can be redispersed in water and the fat removed on a centrifuge or the fat-protein mass added directly to ice cream mix.

(b) *Precipitation of casein* from milk and its subsequent redispersal as a high protein concentrate affords a means of eliminating part or all of the whey constituents from the concentrate. The process should be conducted in such a way as to minimize the denaturation of the casein which occurs in most precipitation methods. Three general types of precipitants have been used, gums, acids and enzymes. If the casein is denatured but its suspension is fine enough to flow through a homogenizer, such treatment will greatly decrease its particle size.

The gum-like precipitants include carrageen or Irish moss, the carob bean or locust pod, pectin, gum tragacanth and gum Karaya. When one of these substances is added to skim milk it causes a slow settling or precipitation of the casein. The whey may then be drained off and a protein concentrate recovered which can be used in ice cream or other food preparations. The decanted whey contains the gum precipitant and it is high in viscosity and difficult to utilize in a condensing or drying operation. Clickner[25] devised a method of reducing the viscosity of whey which contained locust bean gum by digesting it with a mold-produced enzyme.

An early German patent[89] described a process whereby liquid pectin was mixed with skim milk, the mixture was allowed to settle and the whey drained off. In place of pectin Pholmann and Rassers used carrageen[87] while Ambrose[1] and Tracy and Corbett used locust bean gum.[117] Skim milk was pasteurized at 160°F for 20 minutes by Tracy and Corbett. They then sprinkled three ounces of gum into 100 pounds of skim milk with vigorous agitation. When the mixture reached 90°F, or lower during cooling, one pint of a 10 percent solution of calcium chloride was added to aid in obtaining a distinct protein separation. After holding the milk cold and undisturbed for 12 to 48 hours the casein fraction was drawn from the bottom of the vat. This fraction included approximately one-fifth of the original volume of skim milk and contained about 10.1 percent protein, 4.2 percent lactose and 1.5 percent of other solids.

Among the available methods for concentrating casein by acid precipitation several may be cited.[6, 41, 84, 101, 102, 114, 137] These processes consist essentially of precipitation of casein, generally with hydrochloric acid, and redispersal of it with an alkali. The end product in each case is a denatured but edible casein. In the Arbuckle, Shepardson and Walling[6] method the skim milk was coagulated at 95°F with dilute hydrochloric acid. The curd was then broken up and dissolved by adding 5 pounds of sodium bicarbonate per 100 pounds of casein and heating to 150°F for 30 minutes. Enough skim milk was then added to the dissolved curd to produce a finished product of 16 percent solids content, which included 13.8 percent protein, 2.0 percent lactose and 1.5 percent ash.

The usual enzyme precipitant for casein is rennet. Peebles[84] subjected the milk to violent agitation during rennet precipitation and in this way obtained a product with very small granules of curd. Clickner[24] employed a mold enzyme which caused the casein to flocculate as a fine precipitate which could be separated from the whey.

Casein has been redispersed for food uses by digesting it with 1.5 to 7.5 percent of its weight of pancreatin in 2 percent sodium carbonate solution. The digestion was carried out at 104°F for 12 to 16 hours.[106]

**Concentrated Sour Milks.** The process of manufacturing concentrated sour skim milk was developed by Rogers[97] and co-workers[98] to provide a simple and economical method of preserving skim milk solids. Concentrated buttermilk is a product similar to concentrated sour skim milk and is made in much the same way. These products will keep indefinitely if they are not exposed to air. They are used primarily as animal feeds for poultry and hogs.

*Concentrated sour skim milk* is made by developing about 2 percent acidity in skim milk by means of a bulgaricus type culture and a mycoderm, then concentrating the milk to about one-third of its weight. An active culture should be used which is capable of developing an acidity of 1.7 to 2 percent in 18 hours. The "Ga" culture first used for making concentrated sour skim milk was isolated from Swiss cheese whey. The mycoderm was found growing in association with the lactobacillus. Burkey[20] considers that the function or advantage of the associative growth of mycoderm in a culture of lactic acid bacteria is to maintain a more active, viable culture for an extended period of time. The physiological effect of the mycoderm growth on the lactic acid bacteria with which it is associated appears to be either the prevention of the accumulation of excessive acidity or the production of nutritive products favorable for the greater vitality of the bacteria in the culture, or both.

Manufacturing directions for concentrated sour skim milk, according to Rogers,[98] follow: The milk to be concentrated should be pasteurized either in a continuous machine at 170 to 180°F, or by the holding method. A high temperature of pasteurization is desirable, not only to destroy bacteria which might cause abnormal fermentations during the ripening, but also because a high pasteurization temperature tends to make a smoother texture in the finished product. With the holding method this temperature may be 150 to 160°F for 30 minutes.

The vat for ripening should be covered and should have, if possible, a coil for heating, cooling, and stirring. Glass-lined tanks are satisfactory. Cypress vats, if properly constructed and cared for, have been found to give good results. The high acid in conjunction with the relatively high temperature has a tendency to discolor and corrode tinned vats.

When the pasteurized milk is cooled to 115°F, 2 percent of starter should be added slowly with the stirrer running to insure an even distribution. If the milk is not stirred a portion of it will be curdled by the acid, and lumps will form which will injure the quality of the finished product. The starter should be added to the milk. It should not be put in the vat and the sweet milk run into it.

The ripening conditions should be adjusted to allow a drop in temperature of not more than 10° in 18 hours. At the end of this period the acidity of the milk should be between 1.7 and 2 percent lactic acid.

When the sour milk is ready to be concentrated, the curd should be broken up as thoroughly as possible with the ordinary coils or agitators. The milk is then drawn into the pan at the temperature at which it comes from the vat. Forewarming is unnecessary and tends to harden the lumps of curd, making a rough texture in the finished product. Forewarming with direct steam causes the milk to lump so that it cannot be condensed. If the acidity of the milk before concentrating is high enough there is no difficulty from lumping or burning on the coils of the evaporator.

It is essential that the acidity be developed high enough to insure the keeping quality of the finished product. This will vary somewhat with the concentration of the solids, the contamination, and the conditions under which it is held in storage, but 5 percent acidity should be considered the lowest limit. A smoother texture and a more stable product is obtained if the acidity is about 6 percent.

Because of the heavy body of the finished product the degree of concentration cannot be determined by the usual Baumé method. However, three ways of determining the concentration are available. One is the pycnometer method in which a small flask which can be completely filled is weighed on a balance accurate to one-tenth of a gram and this weight compared with that of the same flask filled in the same way with the milk before concentration. The concentration can be easily calculated from the relation of these two weights. The weight which this definite volume of concentrated milk should have will soon be established, and it will be necessary to continue the evaporation only until this weight is reached. A convenient metal flask with attachment for quick filling has been described by Evans.[37] The objection to this method is the difficulty of filling the flask quickly without forming air bubbles which will obviously affect the weight.

A second method is the titrating of the acidity, which increases very nearly in direct relation to the concentration. Thus, if the skim milk contains 9.2 percent solids and a final product with 28 percent solids is required, it will be necessary to concentrate to approximately one-third of the volume. Consequently, if the milk has an acidity of 1.9 percent, the evaporation should be continued until a titration shows an acidity of three times 1.9 percent, or 5.7 percent.

The third method is the determination of the refractive index, which has been suggested for condensed milk,[95] and which is the most satisfactory method for estimating concentration. The refractive index varies directly as the concentration of the soluble solids, and the relation of these constituents to the total solids is nearly constant. A little clear serum is obtained by pressing a small sample of the milk in a cloth with a fine mesh. A few drops of this serum is placed between the plates of the refractometer and the refractive index read. When this is known for a batch of milk with the required amount of total solids, it is then only necessary to continue the evaporation until the refractive index coincides with that of the standard batch.

The concentration should be carried to about 28 percent solids. At this point the concentrate flows freely from the pan and has a thick, pasty consistency when cold. It is run from the pan direct to the container without cooling.

The composition of a concentrated product made from skim milk containing 9.2 percent total solids should be approximately as follows:

| | *Percent* | | *Percent* |
|---|---|---|---|
| Fat | 0.17 | Ash | 2.13 |
| Casein | 8.06 | Lactic acid | 6.08 |
| Albumin | 2.13 | Water | 72.00 |
| Lactose | 9.43 | | |

Whey may be utilized in the manufacture of a concentrated sour product. Equal parts of whey and skim milk are pasteurized and soured separately, then drawn into the vacuum pan and concentrated together. Because of the lower solids content of the whey, such a mixture should be concentrated to a ratio of about 3.9 to 1. The texture and general appearance is in no way inferior to that made from skim milk alone.

*Condensed or semi-solid buttermilk* is creamery buttermilk which is ripened and condensed in much the same way as skim milk. Sour cream buttermilk may be used without subjecting it to an additional ripening period. However, the acidity of the buttermilk before it is condensed should be greater than 1.6 percent as lactic acid if a concentrate of good keeping quality is to be prepared. Sour whey or an organic acid may be used to increase the acidity of buttermilk which is to be condensed for stock feed.

The manufacturing process given for sour skim milk may be followed for buttermilk. Unlike sour skim milk, sour buttermilk should be preheated before it is condensed. Live steam is the simplest heating medium since it simultaneously heats and agitates the mass. Temperatures up to about 160°F may be used for forewarming. The buttermilk should be condensed to about one-third of its weight, giving it a soft gel-like body which will flow readily from the vacuum pan while it is still hot.

*Containers* generally used are oak or fir barrels with a capacity of about 500 pounds. They may be secondhand, but should be tight and coated with paraffin or sodium silicate. It is especially necessary that the barrels be tight, as even a small leak will cause an appreciable loss and may create an air space in which mold will develop. Caution should be observed in storing leaky barrels on a cement floor as the acid whey actively attacks the cement.

Secondhand butter tubs, well paraffined, will serve for local trade but are not satisfactory for shipment. For the customer who wants small lots, tin cans with friction tops are sometimes used; but they rust raipdly and are not entirely satisfactory.

When properly made, the concentrated sour milks will keep indefinitely without appreciable change. If the acidity is above 5 percent, there will be no abnormal fermentations even at summer temperatures, except that when the surface is exposed to the air, molds will develop. They do not freeze at ordinary winter temperatures.

**Whey Concentrates.** Whey is a dilute and highly perishable product, so it is often condensed as plain or sweetened whey to reduce its water content and improve its keeping quality. Plain condensed whey may be used in human or animal food products but the use of sweetened condensed whey is limited to those foods which contain sugar. Whey of any type or quality may be concentrated for use in animal feeds. Acid whey may be partially neutralized with lime before it is condensed, but if the finished product is to be held without refrigeration, the reaction of the concentrate should be below pH 4.5. Acid whey foams less during evaporation in a vacuum pan than does sweet whey. When sweet whey is difficult to manage in the pan, a small quantity of acid or a little fat may be added to break the foam.

Condensed whey has been used in many food products, but successful commercial utilization is limited at present to three important classes of foods—confections, bakery goods and cheese foods. Whey concentrate can also be used in cream-style soups and in whipped dressings and toppings. Other whey-containing foods which as yet have little commercial significance have been developed. When whey is to be used for human foods, it should be cooled or processed at once after removal of the curd.

*Plain condensed whey* is made by pasteurizing the whey and drawing the hot liquid into the vacuum pan. The whey may be condensed to any degree up to a solids content of about 70 percent. Condensed whey of 35 percent and 50 percent solids has been found excellent for chicken feed.[118] Such a whey mixes better with mash than one condensed to a higher solids content. Whey condensed to more than 55 percent solids forms a gel after cooling. This semisolid whey can be used without further preparation for feeding chickens and turkeys.

Density determinations made with a Baumé hydrometer may be used to follow the concentration of the whey in the vacuum pan up to about 64 percent solids. Above this concentration a pycnometer is more accurate than a hydrometer. The relationship between total solids content and the densities of whey is shown in Table 12. When the values in this table are used at temperatures other than 115°F, a temperature correction should be applied. This factor is 0.03 degree Baumé for each degree Fahrenheit by which the temperature of the whey sample deviates from 115°F. This correction factor multiplied by the number of degrees the sample is above 115°F should be added to the Baumé reading given in Table 12 before converting it to percentage of solids. When the temperature of the sample is below 115°F the correction should be subtracted from the Baumé reading. Lawrence[69] in a study of the relation between specific gravity and total solids of whey found that variations in fat from 0.1 to 0.3 percent and in acidity from 0.1 to 0.5 percent had little effect on specific gravity.

TABLE 12. *The Density of Condensed Cheese Whey of Different Percentages of Whey Solids.*[127]

| Total Solids, % | Density at 115°F | | Total Solids, % | Density at 115°F | |
|---|---|---|---|---|---|
| | °Bé. | Sp. gr.* | | °Bé. | Sp. gr.* |
| 30 | 15.6 | 1.121 | 54 | 28.9 | 1.249 |
| 32 | 16.7 | 1.130 | 56 | 30.1 | 1.262 |
| 34 | 17.8 | 1.140 | 58 | 31.2 | 1.274 |
| 36 | 18.9 | 1.150 | 60 | 32.3 | 1.287 |
| 38 | 20.1 | 1.161 | 62 | 33.4 | 1.299 |
| 40 | 21.2 | 1.171 | 64 | 34.5 | 1.312 |
| 42 | 22.3 | 1.182 | 66 | 35.6 | 1.325 |
| 44 | 23.4 | 1.192 | 68 | 36.7 | 1.339 |
| 46 | 24.5 | 1.203 | 70 | 37.8 | 1.353 |
| 48 | 25.6 | 1.214 | 72 | 38.9 | 1.367 |
| 50 | 26.7 | 1.226 | 74 | 40.1 | 1.382 |
| 52 | 27.8 | 1.237 | 76 | 41.2 | 1.397 |

$$^{*}\text{Sp. gr.} = \frac{145}{145 - {}^{\circ}\text{Be.}}$$

Condensed whey is highly supersaturated with lactose, which sometimes starts to crystallize before the batch can be dropped from the pan. At concentrations above 65 percent solids the crystalline structure which forms may cause the product to become so viscous that it will not flow. Paste of this nature must either be dug out of the pan or redissolved by heating it with added water. Crystallization in the pan can be avoided by starting each run in a vacuum pan free from any lactose crystals or insoluble particles. These can induce crystallization as the concentration of lactose increases. Each run should be carried to completion quickly and the batch dropped from the pan into a crystallizing vat or into shipping containers.

A smooth-bodied paste will be produced if the concentrate flows from the pan as a clear sirup, is then rapidly cooled to about 90°F, seeded with lactose and run into cans or barrels. A fairly smooth body may also be obtained by running the concentrate directly into containers, seeding and stirring the whey in each container by hand for about 10 minutes. The formation of minute crystals is favored by rapid crystallization.

The titratable acidity of cheese whey increases approximately in proportion to the increase in concentration as shown in Table 13. Since the high hydrogen-ion concentration of the condensed whey exerts a retarding effect on bacterial growth, plain condensed whey can be shipped or held for a week or two without refrigeration. But when the reaction is pH 4.5 or less and the whey is packed in air-tight barrels or cans so that mold growth is inhibited, it will keep for several months at cool temperatures.

TABLE 13. *Experimental Data Obtained During the Preparation of Five Batches of Condensed Cheese Whey*[127]

| | Plain Whey | | | Condensed Whey | | | | |
|---|---|---|---|---|---|---|---|---|
| Type of Whey | Reaction, pH | Titratable acidity, % | Total solids, % | Reaction, pH | Titratable acidity, % | Density at 115°F | | Total solids, % |
| | | | | | | °Bé. | Sp. gr. | |
| Swiss: | | | | | | | | |
| sweet | 6.12 | 0.11 | 6.64 | 4.75 | 1.14 | 37.6 | 1.350 | 68.39 |
| soured | 4.97 | 0.25 | 6.65 | 4.57 | 2.47 | 37.3 | 1.346 | 73.28 |
| Cheddar: | | | | | | | | |
| sweet | 6.15 | 0.13 | 6.63 | 5.37 | 1.25 | 33.7 | 1.303 | 63.34 |
| soured | 4.44 | 0.36 | 6.77 | 4.18 | 3.86 | 40.1 | 1.382 | 74.65 |
| Cottage | 4.61 | 0.52 | 6.62 | 4.02 | 5.28 | 38.3 | 1.359 | 68.26 |

Meade and Stringham[77] observed that it was necessary to condense whey to about 65 percent solids to produce a firm bodied product but that by special treatment a firm gel could be produced at a solids content as low as 40 percent. Control of the consistency of whey concentrate was desired in the preparation of various animal feeds. The body of the whey concentrate was controlled by mixing two special wheys in the desired proportions. The first was a whey liquor of 45 to 60 percent solids from which about 20 percent of the original lactose had been removed. The second whey was a cultured product in which both lactic and propionic fermentations had been carried out. This whey contained about 6.7 percent total solids, including 1 percent lactic and 0.3 percent propionic acids. When the latter was mixed with the delactosed concentrate the high acidity produced a firm body and the propionates inhibited mold growth. The reaction of the gelled mixture was pH 4.6.

A whey concentrate fortified with protein has been patented by Hall[53] for use as an animal feed. Sour whey of 1 percent acidity is mixed with a protein material such as animal cracklings, waste meat or milk protein. The mixture is concentrated in a vacuum pan to one-tenth its weight. It is stated that the condensation treatment at 145°F for 5 hours also hydrolyzes the protein and thereby improves the nutritive value of the feed.

*Soluble whey protein* has been concentrated for use in food products by Weimar,[132] and by Bell, Peter and Johnson.[14] The protein concentrate is a byproduct of lactose manufacture in this process. Sweet cheese whey of 0.2 percent titratable acidity is neutralized with sodium hydroxide to 0.04 percent acidity (pH 7.3). The neutralized whey is warmed to 140°F and condensed to 32°Baumé at 120°F and a total solids content of about 62 percent. The concentrate is placed in a refrigerator or held in tanks at 32 to 38°F for at least 18 hours with occasional stirring. The crude lactose recovered on a centrifuge contains approximately 91 percent lactose (hydrate), 3 percent protein and ash and 6 percent water. The composition of the mother liquor, which contains the soluble whey protein, is approximately 5.1 percent ash, 14.7 percent soluble protein and 20.5 percent lactose.

The lactoglobulin fraction of milk was suggested[5] as being the constituent largely responsible for the foaming of milk. It is well known that much of the foaming substance of milk is concentrated in the whey. Foam producing material remains in whey even after it has been heated and the coagulated protein removed. The foaming properties of whey protein solutions and their use in salad dressings, and certain candies, icings and custards were investigated by Peter and Bell.[86] However, the mother liquors from any lactose manufacturing process are high in salts and saturated with lactose. Watson,[121] recognizing the limitations caused by the presence of these constituents, attempted to remove them by electrodialysis. More recently, additional investigations on the electrodialysis of whey have been reported.[8, 55, 131] The ash content of fresh whey was reduced from 0.7 to 0.2 percent.[131] A method for the continuous separation of the protein from whey or skim milk by dialysis and gravity stratification has been described.[31] The commercial utilization of soluble whey protein concentrates and dialysed whey preparations has not been extensive. The removal of salts from whey by ion-exchange treatment is considered in Chapter 9.

A foaming agent has been prepared from the mother liquor left from lactose manufacture.[76] The liquor, after removal of the lactose, is diluted with 10 times its weight of water. Half of this mixture is adjusted to pH 7.5 and treated with a flocculating agent (a salt of alginic acid). The treated portion is then mixed with the untreated portion, the mixture is filtered and the clarified filtrate is concentrated to 80 percent solids content. It is claimed that the resultant product will develop a high overrun on whipping and that the foam will set to a stable mass on heating.

*Coagulated whey protein* can be separated from whey for food uses and for the manufacture of protein hydrolyzates. The protein may be precipitated from whey by heat and acid.[21, 22] When cheese whey is used, the separated whey should first be heated to the boiling point in a jacketed kettle or by direct steam jets. Hydrochloric acid is added to attain a reaction of pH 4.8 to 5.3. The whey may also be acidified with other acids such as acetic, phosphoric, lactic or sulfuric. After thoroughly mixing in the acid, the heat is turned off and the whey is allowed to stand undisturbed. The coagulated protein will generally rise and it may be dipped off, or the clear whey drained from the bottom of the vat. If the protein settles to the bottom, the whey may be decanted. The curd may be hung in bags to drain or drainage can proceed from the bottom of a vat, where an outlet strainer is available. When most of the clear whey has been drawn off, the curd is washed well with water to remove soluble impurities. Separation by means of a filter press is possible, but such equipment is not available in the ordinary cheese factory.

The washed, drained curd will contain more than 74 percent moisture, although it will appear quite dry and crumbly. The protein is perishable and may be preserved by drying or freezing. Burkey and Walter[22] found that pressed whey protein would keep several days even at room temperature. Apparently the washed crude protein will not undergo rapid spoilage from bacterial growth, but it will readily support mold growth. This indicates that the curd could be pressed to 65 percent of water, packed in barrels and shipped. It seems improbable that detrimental changes would occur during several days in transit. Perhaps the simplest procedure for preparing the curd is to freeze it and ship it in a frozen condition. Freezing expresses some of the water which cannot be removed by draining.

A whey protein concentrate which has been advocated for use as a beverage, or as a skim milk substitute in food manufacture has been prepared by Smillie and Wight[105] without the use of condensing equipment. Whey is heated to coagulate the albumin, the clarified whey which comprises five-sixths of the total whey is drawn off and the coagulated albumin is redispersed in the remaining whey by homogenization. The product contains about 4 percent whey protein and 10 percent total solids.

The centrifugal separation of denatured protein from whey has been accomplished by means of specially built high speed centrifuges.[32, 104] The whey is first heated to coagulate the protein, then it is centrifuged. The protein fraction is continuously discharged and may vary in moisture content from about 50 to 85 percent, depending upon the kind of machine that is used. Strezynski[112] designed a separator and developed a process[111] whereby a quantity of whey containing 65 pounds of lactose and 6.5 pounds of protein is run through the separator at 150°F to reduce the lactose in the protein fraction to 3 pounds and the protein to 6.3 pounds. A second separator further changes this ratio to 0.15 pounds of lactose to 6.1 pounds of protein. Centrifugal processes of this type are still in the experimental stage and the cost of the centrifuges is too high for any but very large scale installations.

The clarified whey which remains after removal of the protein may be used in lactose manufacture or it may be fed directly to animals. The protein-free whey is often condensed for animal feed. If the feed is to be used at once, the acid whey may be limed to about pH 6.0 to 6.4; if the whey is to be held in storage after condensing, the acid should not be neutralized. The condensation should be carried to 65 to 70 percent solids. The data of Table 12 may be used as an approximate guide to concentrations. Lactose in clarified whey crystallizes faster than it does in plain whey, hence care should be taken not to condense the whey so far that it will solidify in the pan. The heavy mass can be mixed with other feed materials or dried and ground.

*Sweetened condensed whey* was developed[39, 90] to fill the need for an inexpensive and simple method for preserving whey for human food. Separated, pasteurized, sweet cheese whey and a quantity of sugar equal to the weight of solids in the whey (about 6.7 lbs. sugar per 100 lbs. whey) are drawn into a vacuum pan and condensed to at least 76 percent total solids. The specific gravity of sweetened condensed whey at 122°F is 1.360 (38.4° Bé.). The concentrate should be cooled to 95°F, seeded and stirred slowly for one to three hours to crystallize the lactose. It may be packed in barrels or cans and does not require refrigeration during storage.

Sweetened condensed whey darkens and thickens in storage but these changes do not develop to an objectionable degree during the first year. The viscosity increase is less than occurs in sweetened condensed milk held under comparable conditions.

The whipping properties of sweetened condensed whey can be of value in food preparation. The product at room temperature may be whipped to 200 percent overrun in 3 to 4 minutes. The whip will stand without showing drainage for 10 to 15 hours. Uses for whipped sweetened condensed whey include the making of fruit whips, certain candies and frozen dessert preparations. A sweet spread of good keeping quality may be made by mixing equal weights of sweetened condensed whey and peanut butter.

**Miscellaneous Concentrates.** *Concentrated byproduct spreads.* Many attempts have been made to prepare spreads for bread which would be adequate substitutes for butter. None of these has been good enough to remain continuously on the market or to attain a large sales volume. Byproduct spreads may be made by concentrating skim milk, buttermilk or whey to a paste or gel. The mass should have a pleasing flavor and it should have good keeping properties. The concentrate will not spoil readily if it is preserved either by high acid or high sugar content. There is little published information on byproduct spreads and the subject is one which might be profitably investigated.

Sour spreads should have a reaction of pH 4.8 or lower to inhibit bacterial growth. Mold growth may be controlled by air-tight packaging. The high acid will be less objectionable to the taste when the spread is made with relatively weak buffering materials, such as whey solids and vegetable gums, with perhaps a low concentration of casein, than when high concentrations of milk proteins are used. A whey base for such spreads can be readily prepared by allowing cheese whey to sour rapidly to pH 4. The whey is then condensed to 74 percent solids, drawn from the pan, cooled to 95°F, and seeded with stirring for at least half an hour. The reaction of the concentrate will be about pH 4.6. Vegetable gums and various flavoring materials may be added to such a paste.

Substantial quantities of milk fat have been used in some spreads. One such product[136] is described as being made by preparing a concentrate from skim milk and cream, adding 20 percent by weight of cultured buttermilk, 1.3 percent salt and 25 grams of lactic acid per 25 lbs. of total mixture. Starter distillate and vitamin concentrates are added after pasteuerization and the mix is homogenized at sufficient pressure to thicken it slightly. It is filled into containers hot, and then cooled. The spread contains 56 percent moisture, 26 percent milk fat, 16 percent milk solids-not-fat, 1 percent salt and a high vitamin A and D content.

A blend of concentrated whole milk, cream, salt, vegetable gum, acetic acid and artificial flavor and color was produced and sold during World War II as a bread spread. It was slightly acid to the taste and had a mild milk flavor.

Another semi-solid sour spread has been made[48] by coagulating the protein of whole milk, skim milk or buttermilk by acid produced by a lactic starter and by heating the mix to boiling. The fermented, coagulated milk is concentrated under vacuum to various degrees up to about 60 percent solids. The resulting gel may be flavored in various ways including the addition of cured cheese.[49]

Parsons[83] prepared a food product suitable for use as a spread, or as a sandwich filling or salad dressing, when it was mixed with cheese, fats or condiments. A highly concentrated skim or whole milk was heated with stirring until the mixture became brown and attained a roast beef odor. Emulsifying salts were used to make a smooth mixture which could be blended with other foods. Mixtures of this kind would require refrigeration during storage.

Sweet spreads may be prepared by concentrating a low acid milk or whey, generally with sugar. An attractive spread can be made by mixing equal weights of sweetened condensed whey and peanut butter.[128] This mix will keep well at room temperature. A sweet spread made with milk and sugar could be produced as a modified sweetened condensed milk. A spread of this type would resemble some of the Indian sweetened milk products which are produced by cooking milk curd, fat and sugar together. These are discussed in Chapter 9.

A sour spread containing sugar has been prepared to have the following composition: sugar, 37.6 percent; milk fat, 1.15 percent; casein, 4.41 percent; albumin, 1.41 percent; lactose, 7.93 percent; lactic acid, 0.62 percent; ash, 1.4 percent; and water to 100 percent. This product is manufactured by concentrating buttermilk and sugar.[75]

*Block milk* or semi-solid sweetened condensed milk has been manufactured for rations and general purposes in Europe and for confectionery manufacture in the United States. The quantities produced have been small. Block milk is essentially a sweetened condensed whole or skim milk which has a total solids content of 80 to 85 percent and the consistency of soft processed cheese. It is not to be confused with the compressed milk cakes described in Chapter 5 which are made by compression of dried milk.

A method for making solid milk or cream preparations may be outlined on the basis of some of the more practical suggestions of patented processes.[16, 18, 79] The milk product, which may consist of skim or whole milk or cream or mixtures of these products is heated to a temperature between 130 to 150°F. The required quantity of sugar (equivalent to 3.9 lbs. per 100 lbs. of 12 percent solids milk) is added, preferably as a boiled sirup and the batch is evaporated to about 75 percent total solids. The concentrate is then transferred to a small vacuum drying apparatus provided with a suitable stirrer. This second concentrating unit should be heated by a liquid medium rather than direct steam and the stirrer should be capable of thoroughly mixing or kneading the doughy mass. The finished material should have the approximate composition: milk solids 65 percent, sugar

21 percent and water 14 percent. The viscous mass may be packed into fiber containers or formed in blocks, cooled, and coated with a protective material such as cocoa-butter.

A somewhat different approach to the manufacture of block milk is a method by which dried milk is mixed with sugar and water or condensed milk.[43, 44] For example, 20 pounds of sweetened condensed milk is heated to 140°F and then 56 pounds of milk powder and 45 pounds of sugar are added. The creamy mixture is evaporated under vacuum with continuous stirring until the water content is 10 to 15 percent.

The block milk products described above require special agitating and drying equipment to remove the water during the final stages of condensation. A process which could be completed in an ordinary batch type vacuum pan would be of more practical value. This can be done by raising the sugar, and lowering the milk solids and by finishing the batch at 80 to 82 percent total solids. The forewarming treatment previous to evaporation should not exceed 142°F for 30 minutes. The composition of three block milk products is shown in Table 14. Milk 1 is a standard sweetened condensed milk concentrated to 80 percent solids content. This product is suitable for use in candy and bakery products. Products 2 and 3 can be used to form blocks of semi-solid milk and cream.

TABLE 14. *The Composition of Three Block Milk Products*

| Number | M.S.N.F., % | Fat, % | Sugar, % | Water, % | Sugar ratio[1] |
|---|---|---|---|---|---|
| 1 | 36 | 14 | 30 | 20 | 60 |
| 2 | 28 | 12 | 42 | 18 | 70 |
| 3 | 20 | 18 | 44 | 18 | 71 |

[1] Sugar ratio $= \frac{\text{sugar}}{\text{sugar} + \text{water}} \times 100$

The storage stability of the block milks is inferior to that of sweetened condensed milk. There is a tendency, especially at room temperature or above, for the normal thickening which occurs in sweetened condensed milk to proceed to a point where the body of block milk becomes very firm, then finally hard and crumbly. It is difficult to crystallize the lactose properly, consequently some dependence must be placed on the high viscosity of the product to inhibit the growth of large crystals. When the sucrose in water concentration is above 68 percent, sugar crystals may appear. In general, the storage period for this product should not exceed 6 months.

*Condensed ice cream mix.* The service forces used much less condensed ice cream mix during World War II than they did of dry mix. But the condensed mix was a satisfactory product and it has been manufactured for retail outlets by several companies. Condensed ice cream mix may contain between 70 and 80 percent total solids and is actually a paste or jelly preserved by sugar. The composition should be such that when it is diluted with an equal volume of water it is ready to freeze.

The concentrations of the different sugars in condensed ice cream mix affect keeping quality, body, texture and palatability. If the concentration of a normal mix of about 15 percent sugar is doubled, there will be insufficient sugar present in the concentrate to preserve it. When the mix is reduced to a total solids content in excess of 75 percent, it becomes too gelatinous to handle easily. A good condensed mix should have a sugar ratio of 58 to 60, calculated as follows:

$$\frac{\text{sugar}}{\text{sugar} + \text{water}} \times 100 = \text{percent sugar in water or sugar ratio.}$$

This will require about 37 or 38 percent sugar in the finished product.

Dextrose and corn sirup solids improve the body of the frozen ice cream but they promote thickening and color development just as they do in sweetened condensed milk. Dextrose is inferior to sucrose as a preservative. The concentration of solids from corn products should not exceed 25 percent of the total sugar content of the mix.

The lactose concentration of most condensed mixes exceeds that amount which will remain in solution in the water of the mix. The condensed mix should be seeded and cooled with agitation in the same way as sweetened condensed milk. If crystallization of lactose was to be avoided entirely in a concentrate of 26 percent water content, it would probably be necessary to hold the lactose concentration below 3.5 percent. This would allow a milk-solids-not-fat concentration in the finished mix of about 6.8 percent and a lactose in water concentration of 11.9 percent. Data which could be used to establish values for the solubility of lactose at various temperatures and in the presence of the other mix constituents are not available. While the high sucrose content of the mix reduces lactose solubility, the viscous gel structure is effective in retarding crystallization. Concentrated mixes containing up to 8 or 9 percent milk-solids-not-fat probably would not develop lactose crystals during storage but a good mix should contain at least 12 to 14 percent before reconstitution in order to produce a satisfactory frozen ice cream.

The small lactose crystals of concentrated mixes should be thoroughly dissolved during the process of reconstituting the mix with water.

Otting and Quilligan[82] devised a method by which lactose crystallization in concentrated mixes could be suppressed. The fluid skim milk product which was to be used in the mix was treated with a base exchange material to lower its calcium, phosphorus and total ash content. This treatment made it possible to prepare a mix of 74 percent total solids and 12 to 14 percent milk-solids-not-fat in which lactose crystals would not develop during storage. The reason base exchange treated milk suppressed lactose crystallization is unexplained.

Low-lactose concentrates of various types may be employed to increase the protein content of condensed mixes. Otting and Quilligan[82] used an alkali metal caseinate such as that described by Zoller[137] as well as low-lactose milk.[71, 82, 130] The caseinates which improved the whipping properties of the mixes were used in quantities of 0.5 to 5 percent, depending upon the overrun desired in the ice cream. In a mix of 74 percent total solids, Otting and Quilligan[82] used 17 percent milk-solids-not-fat derived as follows: 2.7 percent from the cream, 4 to 6 percent from base exchange treated milk and the remainder from a low-lactose skim milk.

Milk protein is the constituent which is responsible for age thickening in concentrated mixes. The protein in normal milk products appears to cause thickening to a greater extent than that from derived products such as sodium caseinate. Grindrod[50] observed that the casein in concentrated mixes which were treated by his steam injection process redispersed in water more readily than did the casein in mixes heated by other methods. The danger of excessive thickening during the manufacture or storage of condensed mixes makes it advisable to keep the protein content of the mix lower than it is in regular commercial mixes. A very heavy product will result if the milk-solids-not-fat of the condensed mix exceeds 16 or 18 percent.

A concentrated ice cream mix which in some respects resembles the Army paste used during World War II is not difficult to prepare. A suitable formula is:

| | |
|---|---|
| fat | 22.0% |
| m.s.n.f. | 13.5 |
| sucrose | 37.0 |
| egg yolk | 0.4 |
| salt | 0.1 |
| sodium alginate | 1.0 |
| total solids | 74.0% |

This mix without the sugar should be pasteurized at 160°F for 30 minutes, homogenized at 2500 lbs. per sq. in. pressure, then drawn into the vacuum pan followed by the sugar prepared as a boiled sirup. The mix is next condensed to 24.8° Bé. at 120°F or 74 percent total solids content. It is drawn from the pan through sweetened condensed milk coolers or cooled with stirring, as is done for sweetened condensed milk. The product should be carefully packaged to minimize the possibility of contamination.

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# 5

# DRIED PRODUCTS

## INTRODUCTION

Skim milk, whey, buttermilk and other byproducts of milk are dried to reduce volume, to preserve them and to prepare them in a form useful for introduction into concentrated or dried foods. Reduction to the dry state creates problems concerned with solubility, rehydration and keeping quality not encountered in other forms of byproducts.

**Drying Systems.** Conventional methods of drying milk products include processes of three types: shelf or tunnel drying including freeze drying; roller, drum or film drying; and spray drying. The engineering aspects of these processes have been described in detail by Scott[112] and by Hunziker.[59]

*Shelf or tunnel drying* may be considered to include those processes by which the product is first concentrated and then dried in a vacuum or atmospheric shelf or oven type drier. The preliminary concentration is usually carried out by vacuum evaporation as discussed in Chapter 4, CONDENSED PRODUCTS The drying of the concentrate is done either under vacuum or in an atmospheric tunnel drier such as is used for drying casein. Whey and malted milk can be dried by vacuum evaporation followed by tunnel or vacuum oven treatment. A vacuum tray-drying system utilizing radiant heat has been devised.[40]

Freeze drying can be accomplished either by centrifugally removing the ice crystals or by causing them to sublime in a warm vacuum oven. When the ice is separated in a centrifuge the procedure described in Chapter 4 for concentrating milk is followed. The concentrate may be refrozen and reseparated to recover a heavy paste-like mass which must be oven dried under vacuum. One device employs the principle of sudden freezing and drying by injecting the material to be dried into a rotating drum where the small particles are quickly frozen and dried.[39]

Drying by sublimation is done by cooling the product under vacuum until the aqueous portion is frozen, then sustaining the high vacuum while heat is applied to sublime the ice crystals. Protein solutions sublimed from the solid state are not exposed to the high salt concentrations that occur during vacuum evaporation by heat and hence the protein is not so readily denatured. To induce sublimation the vapor pressure at the surface of the material must be lowered below the vapor pressure of ice at 32°F. The drying system must provide for prefreezing the material, a means of maintaining a low vapor-pressure, and a means for supplying the latent heat of evaporation of the ice.[41]

The large volume of water vapor that must be removed is handled by condensation at low temperature or by direct pumping by means of a steam ejector.[38] If a refrigerated surface condenser is used it should have an extensive surface to collect a thin ice film, or it may have a small surface that is continuously de-iced. One system of condensing the water vapor is to spray it with lithium chloride solution. This solution is regenerated by boiling off the water.[15] In its present state of development, freeze drying is too expensive for ordinary byproduct manufacture. But the process is useful where delicate flavors, useful bacteria or critical protein structures must be preserved. Soluble coffee mixed with dried cream is prepared by freeze drying.[66] Drying by sublimation produces an oxygen-free product so that the process is suitable for foods that contain milk fat.

*Roller, film or drum drying* is carried out by coating a single or double drum with the byproduct to be dried, revolving the drums and scraping off the dried material after a partial revolution has been completed. The drums are steam heated and may operate at atmospheric pressure or under vacuum. Skim milk, whey, and buttermilk are usually concentrated in a vacuum pan before they are drum dried. Roller drying can be profitably conducted on a smaller scale than spray drying. Double drum driers occupy only a small space in the plant and can remove 1,000 to 6,000 pounds of water per hour from various types of surplus byproducts.

*Spray drying* is accomplished by spraying the partially concentrated product centrifugally or under 2,000 to 5,000 pounds per square inch hydraulic pressure into a chamber through which a current of heated air is directed. The finely atomized droplets dry almost instantly and the powder is quickly separated from the moistened air. The temperature of the incoming air may vary from 200 to 330°F, but moisture evaporation from each droplet keeps the powder particles relatively cool and the milk protein is not denatured. Drying units usually handle 30,000 to 150,000 pounds of fluid byproduct daily with an hourly production of 200 to 1,000 pounds of powder. In heavy cheese-producing areas whey may be trucked to a central condensery. Such a plant will sometimes handle 1,500,000 pounds of whey daily, at a rate as high as 5,000 pounds of dried whey per hour.[121]

Two types of spray drying plants are common in the United States. With one system the milk or whey product is sprayed into a large rectangular chamber where on meeting heated air it dries and falls to the floor. The moisture-laden air escapes through a dust collection system which generally consists of a large number of filter bags. The dried product may be continuously removed from the floor of the drier or it may be collected at the end of the run. The Merrell-Soule, C. E. Rogers, and George Scott & Son driers employ this process. A new Scott installation in England has recently been described.[5]

The second type of plant dries the product as it is sprayed into a cyclone of heated air. The powder is removed from the bottom of a cone-shaped drying chamber and the fine particles are separated from the air by means of one or more cyclone collectors. The last traces of powder may be washed from the exhaust air in a concentrator, by a spray composed of fluid product coming into the system. Precondensing is thus done within the system by exhaust air. This is the Gray-Jensen process.

## TECHNICAL PROBLEMS

In the manufacture of the dried byproducts of milk the technical problems that determine and control production methods, quality, and consumer utilization fall into three groups: byproduct processing before drying, byproduct problems associated with the drying process, and storage changes.

**Processing before Drying.** The predrying treatment of byproducts includes a preliminary heat treatment, adjustment of composition by the addition or removal of certain constituents, homogenization of special products, and concentration.

*Preheating* byproducts before drying is necessary to destroy microorganisms and enzymes and to control the physicochemical condition of the milk constituents. The heat treatment must be at least equivalent to pasteurization so that undesirable organisms will be destroyed. It is very important that the rapidly multiplying bacteria in cheese whey or any other cultured product be destroyed as soon as possible to retard excessive development of acid and off-flavors.

The enzyme lipase, present in milk, splits milk fat by hydrolysis and the resulting free fatty acids produce rancid odors and flavors. Since most byproducts are almost fat-free, lipase does not cause off-flavors when they are dried. However, complete destruction of lipase in those products that contain considerable quantities of fat must be accomplished before drying. Traces of enzyme remaining in the dried product may act slowly during long periods of storage. This cannot occur in pasteurized, perishable, refrigerated goods which are consumed before they can be injured by enzyme action. Available evidence[44, 70, 107] indicates that heating to at least 176°F for 15 minutes but preferably within the range 185 to 195°F for 15 minutes is necessary to inactivate the lipase. Sugar in the mixture being heated protects the enzyme and a temperature of at least 180°F for 15 minutes is necessary to prevent lipolytic action in sweetened condensed milk.[105]

Other enzymes, proteases, peroxidases, and catalases may be present in byproducts but they are largely destroyed during the usual preheating treatment. Traces of these enzymes remaining in the dried products do not seem to be active enough to produce undesirable changes during normal periods of storage.

The type of preheating treatment given to skim milk before it is dried is governed by the requirements of the food in which the dried product is to be used. Skim milk is preheated either for use in various foods, including ice cream, or for bakery goods. The preheating treatment for general use is 160 to 165°F for 30 minutes while for bakery use it is above 180°F for 30 minutes.

Greenbank et al.[43] first showed that skim milk heated to 185 to 203°F for ½-hour and subsequently condensed and dried, had better baking quality in bread than when lesser heat treatments were used. The viscosities of the powders reconstituted to 40 per cent solids were much greater in the samples that were heated above 185°F than were the viscosities of the samples heated to lower temperatures. Some effects of preheating milk and whey products on bakery goods are discussed in Chapter 7.

The solubility of dried milk is not greatly affected by differences in raw milk preheating temperatures up to about 190°F. The tendency is toward a decrease in solubility as the preheating temperature is raised.[27, 28, 78] However, at temperatures above 190°F there is some indication (Sample 12-27-44, Table 16) that the stabilizing effects of high temperatures increase protein stability and improve solubility of the powder particles. The data should be extended to show the effects of forewarming at temperatures above 212°F. The effect of preheating concentrated skim milk upon the solubility of its dried product is discussed later in this section.

Preheating concentrated skim milk to temperatures above boiling by means of high-temperature short-time heaters has recently attracted the interest of many processors. A discussion of high-velocity high-temperature heaters appears in Chapter 8. When concentrated skim milk is preheated to above boiling temperatures before it is dried, it explodes into a mist in the drying chamber. This aids atomization and increases drier capacity. Little is known as to whether the short exposure at high temperatures imparts beneficial characteristics to the powder that cannot be obtained at below boiling temperatures. It is certain that the physical state of the protein can be altered since coagulation is within easy reach at high concentrations and high temperatures.

When the coagulation of a concentrated skim milk is approached the development of excessive viscosity may increase pumping and handling difficulties and be otherwise objectionable. It is therefore of interest to study the relationships among the conditions of heating, coagulation and viscosity development.

It has been shown[32] that the viscosity of concentrated milks increases rapidly just prior to coagulation. Maximum viscosity is attained shortly after coagulation but before extrusion of the whey. Concentrated milks of high heat stability exhibit a lower viscosity before coagulation than less stable milks. Destabilizing effects such as aging, addition of lactic acid or low forewarming temperatures applied before concentration produce higher viscosity values in the concentrates at the time of coagulation than when these effects are absent.

TABLE 15. *The Relationship between the Concentration, Viscosity and Heat Stability of Skim Milk*[1] *Heated to 185°F after Concentration.*[140]

| Concentration S.N.F. percent | Viscosity at 86°F | | | Heat Stability at 185°F, min. |
|---|---|---|---|---|
| | Before Heating at 185°F, poises | After Heating to 185° F, 10 minutes, poises | At Coagulation, poises | |
| 17.88 | 0.06 | 0.06 | 2.01 | 1920 |
| 26.63 | 0.14 | 0.34 | 7.75 | 140 |
| 35.04 | 0.56 | 24.30 | 24.30 | 10 |
| 37.45 | 0.96 | 314.16 | 30.38 | 7 |
| 38.86 | 1.55 | 607.50 | 43.40 | 5 |
| 41.72 | 3.29 | 1,428.00 | 57.66 | 2 |

[1] Procedure: the skim milk was forewarmed to 185°F, 10 minutes, condensed under vacuum to 41.72 percent solids, cooled and diluted with water to the lower concentrations. The various samples were then heated as indicated in small cans in a pilot sterilizer, reel revolving at 4 r.p.m.

The relationship between the concentration, viscosity and heat stability of skim milk heated in cans with very mild agitation is shown in Table 15. Skim milks of more than 40 per cent solids are fluid and easily handled if they are not heated and held at high temperatures. The 41 per cent skim milk of Table 15 began to coagulate after it was held without agitation at 185°F for two minutes; it was a firm gel after 10 minutes at this temperature.

Agitation during heating can be used to retard the development of excessive viscosity in concentrated milks. The relationship between viscosity and heat treatment of concentrated skim milks that were subjected to different types of agitation is shown in Figure 8. Homogenization destroyed viscosity but the violent agitation that accompanied heating in a high velocity tubular heater prevented even the formation of a viscous structure. The slight rise in viscosity in the lower curve between 272 and 278°F is in sharp contrast to the steep rise in the upper curve. Yet both milks passed through the coagulation phase and both finally showed the beginning of wheying off with an accompanying fall in viscosity.

The solubility of the dried product is lessened if the concentrate is subjected to any heat treatment that may cause incipient coagulation. If

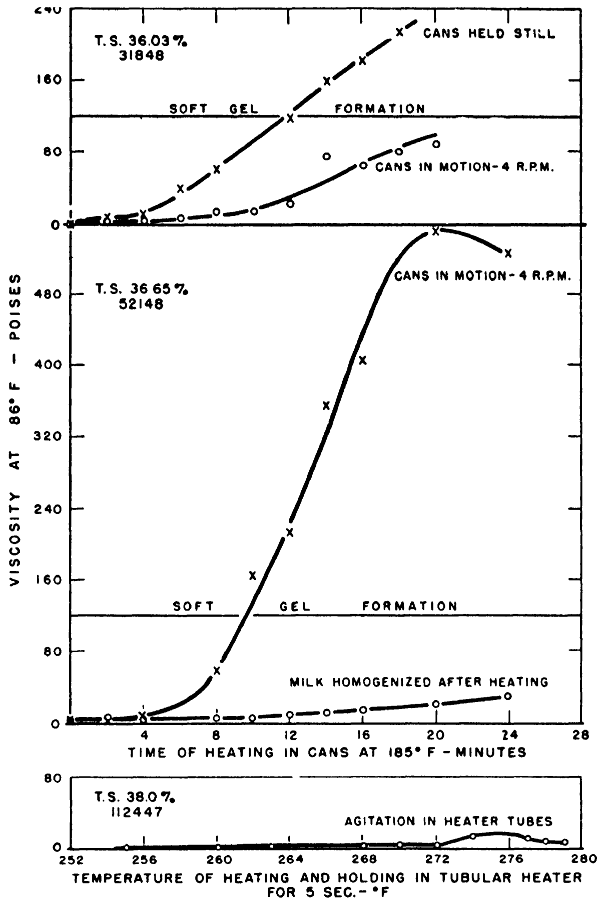


Fig. 8. The effect of different types of agitation during heating on the viscosities of three concentrated skim milks.[140]

a dried skim milk of high solubility is required, the protein of the condensed skim milk must not approach the coagulation point before it is sprayed. The coagulation data of Chapter 8 indicate that holding one minute at 220, 230, 240, 250, and 260°F will cause skim milks of 48½, 43, 38, 32, and 28 per cent solids, respectively, to coagulate.

The effect of heat treatment of skim milk both before and after it is concentrated for drying, on the solubility of its spray dried product, is shown in Table 16. The low solubility of sample 12-1-44 was caused by heating it to 180°F after it had been concentrated to 38 per cent solids content. The results of similar experiments conducted at preheating temperatures above boiling are not yet available.

Peebles and Manning[98] found that when whey was preheated until the proteins coagulated, the precipitated protein did not interfere with lactose crystal formation during the drying process, but it did help to produce a stable, non-hygroscopic dried whey.

TABLE 16. *Effect of Conditions of Manufacture on the Solubility of Dried Skim Milk Manufactured by the Gray-Jensen Process, without the Use of the Wet Collector.*[1]

| Sample number | Heat Treatment before Condensing in Vacuum Pan | | Solids Content when Sprayed % | Heat Treatment before Spraying | | Drier Air Temperature | | Tests on Fresh Powder | |
|---|---|---|---|---|---|---|---|---|---|
| | Temp., °F | Time, min. | | Temp. °F | Time, min. | In, °F | Out, °F | Moisture % | Solubility, c.c.[2] |
| 11/23/44 | 180 | 10 | 38 | 122 | 0 | 257 | 208 | 3.52 | 0.3 |
| 12/1/44 | 180 | 10 | 38 | 180 | 0 | 282 | 240 | 2.21 | 3.12 |
| | 180 | 10 | 38 | 180 | 40 | 282 | 240 | 3.56 | 10.0 |
| 10/14/44 | Not condensed | | 9 | 180 | 10 | 262 | 212 | — | 0.2 |
| 12/14/44 | 175 | 30 | 28 | 176 | 10 | 282 | 230 | 1.0 | 0.3 |
| | 175 | 30 | 32 | 176 | 10 | 282 | 230 | 1.05 | 0.42 |
| | 175 | 30 | 36 | 176 | 10 | 282 | 230 | 1.91 | 0.63 |
| | 175 | 30 | 28 | 176 | 10 | 240 | 175 | 2.78 | 0.10 |
| | 175 | 30 | 32 | 176 | 10 | 240 | 175 | 2.93 | 0.10 |
| | 175 | 30 | 36 | 176 | 10 | 240 | 175 | 3.68 | 0.10 |
| 12/27/44 | 175 | 30 | 39.30 | 170 | 0 | 282 | 230 | 2.32 | 1.2 |
| | 190 | 30 | 39.97 | 170 | 0 | 282 | 230 | 2.46 | 1.1 |
| | 205 | 30 | 40.20 | 170 | 0 | 282 | 230 | 2.33 | 0.7 |
| 1/5/45 | 175 | 30 | 41.09 | 160 | 10 | 283 | 230 | 2.91 | 0.65 |
| | 175 | 30 | 41.09 | 160 | 10 | 266 | 210 | 3.87 | 0.15 |

[1] Acidity of all reconstituted powders was 0.14 to 0.15% except that those of 12/1/44 = 0.19% and 10/14/44 = 0.16% titratable acidity.

[2] Solubility tests made according to the American Dry Milk Institute method. Data from unpublished work.[138]

*Composition adjustment* is carried out before drying to improve the drying characteristics of the byproduct and to manufacture special products. The adjustment may be made in either the soluble or the colloidal constituents of the mixture to be dried. An increase in soluble substances such as sugars, acids, or salts makes the material more difficult to dry than when the concentration of these substances is decreased. The addition of colloids or suspended matter such as proteins, cereals, stabilizers and drying aids is helpful in the drying process. Whey is more difficult to dry than skim milk because of its low protein and high lactose content.

Lactic acid development in skim milk, whey and buttermilk should be retarded by proper pasteurization and cold storage of the products during manufacture and before drying. When the reaction of a product is more acid than pH 6, it should be neutralized before it is dried. Skim milk that has been properly cared for will need no neutralization. Sour cream is neutralized before pasteurization and churning but the resulting buttermilk is high in lactates. This causes the buttermilk to be unusually difficult to dry and makes the powder very hygroscopic. Waite[134] found that acid casein whey and some cheese wheys required neutralization before they could be drum dried. Calcium hydroxide was used to reduce the acidity from 0.48 percent to 0.18 percent but this treatment reduced the palatability of the dried whey. Lavett[75] adjusted the titratable acidity of whey to between 0.30 percent and 0.40 percent before he dried it on a single pair of rolls.

Drying aids may be added, especially to whey, to improve drying characteristics. Pectic acid has been used in the drying of mixtures of whey and fruit juices.[147] The dried whey can be scraped off the rollers easier if a finely divided dry material such as dried whey is sprinkled on the semi-dried film as the rolls revolve.[8] It has been found[63, 116, 122] that whey dried with less difficulty when it was mixed with a material which formed a sheet as it was scraped from the drums. Skim milk, buttermilk and organic, water-insoluble, non-gelatinized substances such as ground and sifted cereals were suitable drying aids. Stevens[118] demonstrated that a mixture consisting of equal parts of potato and whey solids could be easily dried on a drum drier.

Casein sometimes is removed from skim milk by treatment with one of several gums.[2] The resulting whey is highly viscous; it may be treated with an enzyme that will act on the gum, thus reducing the viscosity of the whey and making it easier to handle in drying equipment.[21]

The composition of special products is sometimes adjusted by the addition of sugar, a substance that always increases both drying difficulties and hygroscopicity of the dried material. However, skim milk with a sugar content as high as that present in sweetened condensed skim milk (30 percent S.N.F. and 42 percent sugar) can be dried. Even molasses has been spray dried for use in prepared gingerbread mixes. When foods of this character are spray dried it is essential that the drying temperatures be high enough to quickly and completely dry the material so that stickiness will be reduced to a minimum. Rapid movement and cooling of the dried particles in the collection system will reduce the caking tendency.

Malted milk is made by the addition of an extract of barley malt and wheat flour to whole milk so that the composition of the finished product will be 7.5 percent fat.

Adjustment in composition before drying permits the manufacture of a wide variety of byproducts.

*Homogenization* is often used to disperse the fat in some products before they are dried. Ice cream mix or other materials that contain fat should be homogenized, especially in those cases where the fat is added to the mixture as butter oil or frozen and thawed cream. The high pressure spraying that precedes spray drying is in itself a mild homogenization treatment, being equivalent to homogenization at 500 to 1,000 pounds per square inch pressure.

Homogenization is useful for dispersing insoluble protein and other constituents of special byproducts such as soups that are to be spray dried. Various types of mills and grinding equipment often serve to physically prepare the body of a product for drying.

Homogenization appears to help to protect the fat in dried products from oxidation. In the case of dried whole milk this protection was attributed by Holm, Greenbank and Deysher[55] to a better coverage of the fat by adsorbed protein in the homogenized product. They found that, as the fat content of a powder was raised, the amount of free fat increased sharply at 22 percent, whereas when the product was homogenized before drying a sharp rise in free fat did not take place until the fat content of the powder reached 26 percent. The large quantity of free fat in an unhomogenized dried milk of 26 percent fat made it quickly susceptible to oxidation.

*Concentration* before drying is practiced to increase the capacity of the drying equipment, to reduce drying costs and, for spray drying, to control the size and oxygen content of the powder particle.

Concentration of skim milk to 30 percent solids increased the capacity of an atmospheric drum drier 26 percent over its capacity to dry plain skim milk, according to calculations from the data of Combs and Hubbard.[23] Other estimates by manufacturers of drum driers indicate that drier capacity can be increased as much as 70 percent if the skim milk is precondensed. Since there is less water to remove from concentrated skim milk a thicker film can be formed on the drum surface than when fluid skim milk is used.

Despite the advantages of drying at high concentrations and high temperatures, certain limits must be observed. The product must be concentrated and handled so that the formation of large lactose crystals that might obstruct spray openings and damage pumps will be avoided. Most byproducts can be dried over a range of concentrations of 30 percent to 55 percent solids. The excessive heating at high concentrations which produces thickening and coagulation in skim milk and results in drying difficulties and insoluble powder has been discussed earlier in this chapter.

Drying costs are reduced by precondensing because water remov alunder vacuum is more efficient than under atmospheric pressure and becaues plant output is greatly increased when a concentrate is dried.

The particle size of spray powder increases as the solids content of the concentrate being dried is raised. Powder containing fat keeps better when it is made from concentrated milk than when it is sprayed from unconcentrated milk,[55] apparently because the particle from concentrated milk is larger and contains relatively less oxygen than that from unconcentrated milk.[46]

*The bacteriological quality* of spray dried milk was found by Mattick, Hiscox and Crossley[79] to be determined by three factors listed in order of importance; plant cleanliness, preheating temperature, and bacteriological quality of the raw milk. Detailed information on the bacteriology of dried milk and milk byproducts may be found in the papers of several British workers.[28, 49, 50, 79, 87, 142] The plate count of a powder can of course be lowered by the use of high preheating temperatures before drying. Thermophilic organisms may be a source of high counts in powders and they may cause the count to increase during a continuous day's run. The plate counts of both roller and spray dried powders tend to decrease during storage.

**Behavior of the Byproduct Constituents during Drying.** Byproduct problems associated with the drying process are concerned largely with the two constituents, protein and lactose. The physical condition of both protein and lactose must be controlled when mixtures are dried for special purposes. The concentration of ingredients in the products to be dried and the time and temperature of exposure to drying conditions are of great importance in determining the characteristics of the dried material.

*Protein* coagulation occurs during atmospheric roller drying but it can be avoided in the spray process. Coagulation of the whey protein fraction is often brought about to prepare dried products for special purposes such as the conditioning of skim milk for bakery use. (See Chapter 7.) It is important to prevent coagulation of the casein during drying if a readily dispersible and soluble powder is to be prepared.

The fundamental differences between the effect of roller and spray drying on the casein of milk lie in the time-temperature-concentration relationships that are characteristic of the two processes. The lowest temperature to which the roller dried product is subjected as it boils between the drums is 212°F. As the film revolves on the hot rolls, the concentration of its constituents increases and this increases its boiling point. The progressively rising protein concentration causes a rapid decrease in the resistance of the calcium caseinate system to coagulation. Finally at the high temperature and concentration that exists in the fraction of a second before the film is dried and removed, complete protein denaturation occurs.

In contrast, spray dried products are high in solubility because the continuous cooling of each small droplet by the vaporizing water prevents overheating. Drying is almost instantaneous in the spray process and the protein is not rapidly coagulated in the dry state. Time can be taken to collect the spray dried particles without denaturing them. Wright[149] was the first to show the effect of the time-temperature-concentration relationship on the solubility of roller dried milk. Figure 9 has been drawn from his data. It shows that the calcium caseinate complex is rapidly denatured in highly concentrated milk (13.34 percent moisture) and that it is much more resistant in the dry state (2.44 percent moisture). Milk that reached a concentration of 86 percent solids and a temperature of 230°F during the final stages of drying on a hot roll became 50 percent insoluble after $\frac{1}{10}$-second exposure. In contrast, a sprayed droplet of milk instantaneously dried to 2.4 percent moisture could be heated to 340°F for $\frac{1}{10}$-second before it showed 50 percent insolubility. Some of these time-temperature-concentration relationships are discussed in the preheating section of this chapter and in Chapter 8.

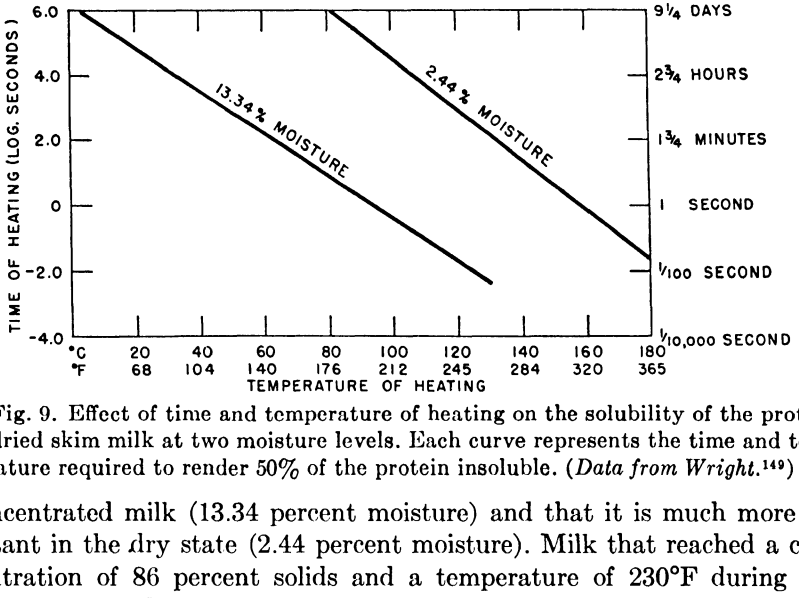


Fig. 9. Effect of time and temperature of heating on the solubility of the protein in dried skim milk at two moisture levels. Each curve represents the time and temperature required to render 50% of the protein insoluble. (*Data from Wright.*[149])

Howat and Wright[57] found the solubility of roller dried milk varied with the temperature of reconstitution. Maximum solubility occurred at 122°F and the protein that was insoluble at this temperature (about 25 percent of total protein) was considered to have been denatured by moist heating during drying. There was an increase in the amount of insoluble protein to at least 50 percent of the total protein when the powder was reconstituted at 68°F. This added increment of insoluble protein was found to be caused by the effect of dry heating of the powder during the final stages of the roller drying process.

When roller drying is carried out under a vacuum, overheating of the protein is avoided and the resulting dried product shows only slightly less solubility than the spray dried material. A vacuum treatment to lower the moisture and improve the solubility of spray dried powder has been proposed.[113] The milk is dried so that it contains a higher moisture content than that desired in the finished product. The hot powder is conveyed to a vacuum chamber where part of the remaining moisture is flashed off. The powder is then cooled.

Protein tends to make a byproduct easy to dry in contrast to difficulties that are encountered when the material is high in lactose or salts. As the protein content of skim milk is lowered it becomes more difficult to dry. When all the casein is removed, the resulting whey dries as a sticky, hygroscopic mass. The drying characteristics of whey can be improved by heat coagulating the whey protein before drying the product.[98] Similarly, recent evidence[120] indicates that if the casein of a byproduct mixture is made insoluble by rennet, acid or heat, the mechanical difficulties of drying a normally sticky product are lessened. However, it appears to be more difficult to remove the last water or to obtain as dry a powder from a skim milk that contains coagulated casein than from one that contains undenatured casein.

The temperature of the air in a spray drying unit is shown in Table 16 to affect the solubility of the dried product. When inlet air was 280°F and outlet air 230°F, the solubility of the dried skim milk was poorer than when these temperatures were lower. The outlet air especially, should be maintained at as low a value as it is possible to use to produce a powder not greater in moisture than 3 percent to 4 percent.

*Lactose*, present as a concentrated sirup or glass, was first shown by Troy and Sharp[131] to be the cause of caking of dried milk and whey. The lactose solution is concentrated in these dried products to such a degree that it cannot crystallize as long as the moisture content of the powder is maintained at a low level. But the concentrated lactose sirup is very hygroscopic and it quickly absorbs water when exposed to moist atmospheres. Water dilutes the sirup and causes crystallization of $\alpha$-lactose hydrate with one molecule of water. The crystal structure that forms causes caking of the product. Many of the patented processes for drying dairy byproducts and especially whey were devised principally to remedy this difficulty of lactose crystallization and caking.

The formation of a lactose glass during drying is more serious in the case of whey than skim milk because three quarters of the solids of whey is lactose while only one half of the solids of skim milk is lactose. Whey tends to form a more hygroscopic and sticky product than skim milk so that most of the special modifications in the drying process are designed for whey drying.

Processes for controlling the crystallization of lactose during the manufacture of products that contain 60 percent to 80 percent lactose may be divided into four groups. (a) The byproduct, generally whey, is concentrated to 70 percent solids or more, the lactose is allowed to crystallize, then the mass is dried.[12, 68, 74, 114] (b) The byproduct is dried and then it is allowed to absorb water to force the crystallization of the lactose.[19, 34, 96, 136] Finely divided powder may be dispersed in a moist atmosphere and after lactose crystallization is complete excess water is removed in a final drying process. (c) The byproduct is concentrated to the point at which it contains approximately the quantity of water required for hydration of the lactose.[8, 72, 73, 94, 97] No water is added and under carefully controlled conditions it is not necessary to remove water after the crystals have formed. In practice, however, often about 2 percent to 5 percent of water must be removed after crystallization of the lactose is complete. (d) A dried product in which the lactose is largely in the $\beta$-anhydride form is prepared by seeding a partially concentrated product such as whey with $\beta$-lactose at a temperature above 200°F and holding it at this temperature while it crystallizes and dries.[10, 19, 73]

*Other constituents* of milk or materials added to byproducts before drying affect the ease with which various mixtures can be reduced to dryness. In general, mixtures of proteinaceous and starchy materials have good drying characteristics while the presence of milk salts, lactic or other acids and the sugars make drying difficult. A method of classifying materials according to the ease with which they could be dried might be based upon their molecular size. Proteins of large molecular size, such as the calcium caseinate complex, dry easier than suspensions of the smaller whey protein molecules. The heat denaturation of protein which increases its molecular size also improves its drying characteristics. The hexose sugars and the disaccharides dry initially to an amorphous glass but the molecularly larger starch suspensions may be dried easily on drum or spray equipment.

Fatty materials dry readily if mixed with protein or starch. However, fat generally is absent, or present only in small quantities, in most byproducts. When fat concentrations are high the dried material is difficult to collect and handle because the low melting fats remain liquid during drying operations. If fat emulsions are to be dried it is necessary to provide sufficient protein or other surface active material to protect each fat globule with an adsorbed membrane. The emulsion can be stabilized by homogenization but an adequate supply of membrane material must be provided to cover the new fat surfaces created during homogenization. Lacking the required quantity of protective material homogenization and even high pressure spraying can readily break an emulsion and release free fat. Studies on the freezing and thawing of homogenized cream[139] indicate that the new fat surfaces created by homogenization require protein for protective coverage to the extent of at least 10 percent of the weight of the fat in the cream. One continuous buttermaking process breaks 80 percent fat (cream) emulsions by homogenization. Fatty mixtures to be homogenized and spray dried should be adjusted so they contain no more fat than ten times their protein content.

**Storage Changes in Dried Byproducts.** The physical and chemical changes that occur in dried byproducts during storage are associated with the three important milk constituents; fat, protein, and lactose.

*Milk fat changes.* Milk fat generally is not present in significant quantities in byproducts, but special foods containing fat are sometimes made and among these there are fat spoilage problems. Deterioration in milk fat is of two types: rancidity, which is the result of hydrolytic changes, and tallowiness, produced by oxidation of the fat. These changes have been discussed by Holm and Greenbank[54] and more recently reviewed by Greenbank.[42] Much work has been done on the deterioration of milk fat and no attempt will be made to include a detailed account of it in this discussion on byproducts.

The rancidity that develops in some dried products is generally caused by a trace of lipase that has not been destroyed in the predrying heat treatment. The temperature-time relationships for the destruction of lipase have been considered in this chapter under *Preheating.*

The rate of oxidation of the fat in a dried byproduct depends partly upon the quality of the original milk or milk product from which the dried material was prepared. These products should be low in bacterial content and they should be dried as soon after the original milk is drawn from the cow as possible. The presence of metal catalysts, especially copper and iron, greatly accelerates the rate of oxidation. Consequently, all equipment with which the byproduct comes in contact during the course of its processing should be of stainless steel.

Low oxygen concentrations in the atmosphere of the container reduce the rate of deterioration of the powder when milk fat is present. The dried product should be packaged in cans and the oxygen level reduced to about 2 percent of the gas in the container. It is customary to replace the air removed from the can with nitrogen. A single evacuation at about 5 mm. pressure for 10 to 15 minutes will reduce the oxygen content to 3 percent. A double evacuation and filling with inert gas with a holding period between evacuations of 4 to 7 days will produce a concentration of about 2 percent oxygen in the atmosphere of the container, a percentage which will not rise above 3 percent by release of adsorbed oxygen during storage.[53]

The rate of deterioration of fat practically doubles for each increase of 18°F in the temperature of storage.[53]

Since the fat content of dried byproducts may vary widely and since it may not always be possible to control the freshness of all the constituents of a mixture, the specific requirements for producing a product which will not develop oxidized flavor or tallowiness must be judged separately for each byproduct. Dried buttermilk, for example, is very susceptible to spoilage by oxidation of its fats and phospholipids. The constituents of buttermilk are usually subjected to more severe oxidative conditions than are the components of milk that is dried soon after production.

*Protein and lactose changes.* The reaction between milk protein and lactose in the dry state causes deterioration in the color, flavor and solubility of dried milk byproducts during storage. High moisture content of the powders and high storage temperatures accelerate the reaction.

The discoloration is very rapid during storage above 85°F and at relative humidities above 30 percent. The reaction rate is slow when the moisture content of powders remains below 2.5 percent. Kass[65] has studied the browning of dairy products and summarizes the characteristics of the reaction as follows: "The browning reaction is a complex function of time and temperature, and at certain stages appears to be autocatalytic. It is characterized by the development of acidity, fall in pH and oxidation-reduction potential, comparatively slight loss of copper-reducing ability, an appreciable but constant conversion of aldoses to ketoses and pronounced loss of optical activity. The coloration is a logarithmic function of the amount of lactose optically inactivated, the latter being directly proportional to the initial concentration. The coloring matter is adsorbed by the casein, but the discoloration cannot be directly related to the formol titer or the amino-nitrogen, and the racemization curves of casein precipitated from raw and browned milks are identical." Doob, Willmann, and Sharp[33] working with both dried skim milk and dried whey showed that browning during storage was associated with osmotically held moisture, particularly with water other than that present in $\alpha$-lactose hydrate. Similarly, recent studies of storage changes in dried whole and skim milks have shown that the browning reaction is accelerated with increases in the moisture level.[26, 48, 52, 69, 124]

A method for the determination of the color of the dry products of milk has recently been described.[18]

It is well recognized that the browning of foods involves many reactions. Trace metals have been suspected of catalyzing browning and it has been shown that trace amounts of metals, particularly copper, accelerate the rate of browning.[7] Reactions of copper and proteins have been described and it is indicated that copper-protein complexes can promote browning of a different kind from the usual types of non-enzymatic browning.[128] Powder made from sour buttermilk shows a greater affinity for moisture and discolors faster than powder made from sweet buttermilk.[30]

Lea[76] found that the reducing sugars combined with free amino-groups of milk protein in a 1:1 ratio to produce insolubility as well as discoloration. Sucrose did not enter into such a combination and glucose reacted more rapidly than lactose. Sucrose and lactose delayed the onset of glucose-induced insolubility but they did not prevent the discoloration. Lea's work indicates that the development of protein insolubility and discoloration during storage of dried milk byproducts will be accelerated by the presence of glucose. The glucose-induced changes could probably be retarded by inclusion of sucrose in the formula.

The deterioration in dried milk byproducts during storage may be illustrated by some of the data of Henry et al.[48] obtained in an extensive study of dried skim milk. Figure 10 was prepared from their results. It shows that rapid deterioration in color, flavor and solubility can be expected if powders of more than 5 percent moisture are held at temperatures above 83°F. Earlier[123] it had been observed that milk powder became progressively less soluble during storage when its moisture content exceeded 3 percent.

The nature of the changes that take place when dried milks deteriorate during storage is not clear. Henry et al.[48] considered the changes to originate from a reaction between the protein amino-groups and lactose. This reaction, which had a high temperature coefficient, took place in at least two stages. The primary combination resulted neither in discoloration nor in loss of solubility. These followed only as a result of secondary changes, the nature of which was not fully understood. Crowe et al.[29] showed that some of the reducing substances produced both by the heat treatment of milk and by the aging of dried milk were formed from lactose and from interactions of protein with lactose. Coulter, Jenness and Crowe[26] summarize the non-lipid changes that occur in dried whole milk in storage as follows: "development of a stale or burnt-feathers flavor, production of acid ferricyanide and indophenol reducing substances, production of carbon dioxide, utilization of oxygen, production of water, production of extractable fluorescent materials, browning, loss of lactose, increase in acidity and loss in protein solubility." All of these changes were found to increase in rate with increase in moisture content and temperature. Table 17 shows the storage changes in dried skim milk of high moisture as described by Henry et al.[47]

Although moisture is harmful to the solubility of a powder during storage, some processes permit moisture absorption in order to facilitate lactose crystallization. Chuck[19] discovered that when dried milks were quickly exposed to finely atomized water which promoted rapid lactose crystallization, and when the excess water was promptly removed, the amount of protein rendered insoluble was less than 2 percent. The powders did not

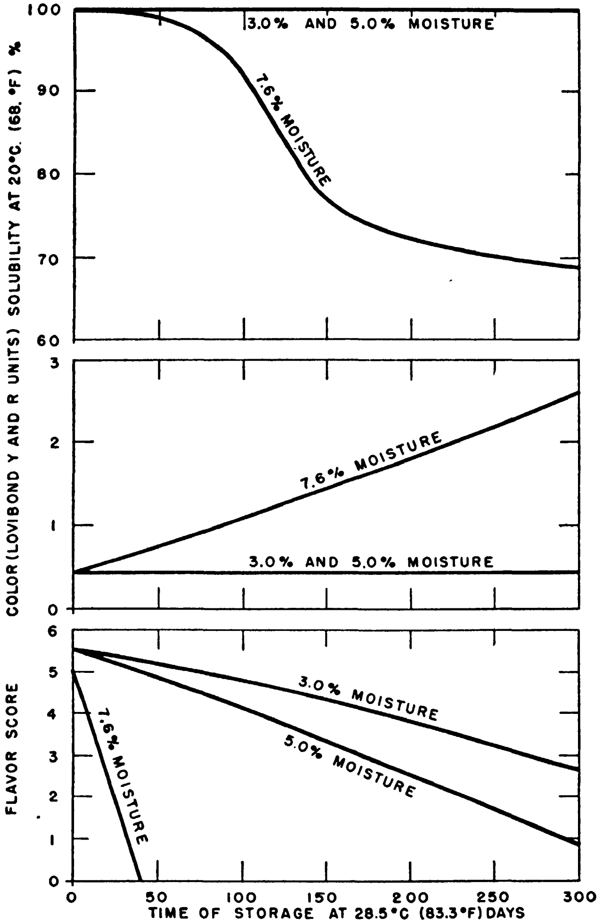


Fig. 10. The effects of differences in moisture content on flavor, color and solubility changes in dried skim milk during storage. (*From data of Henry, Kon, Lea and White.*[48])

subsequently cake when exposed to moisture. The percentage protein rendered insoluble as a result of caking was: dried skim milk, 85 percent; dried buttermilk, 84 percent; and dried whey, 55 percent. The greater loss of protein solubility in skim milk and buttermilk than in whey was attributed to the high casein content of the milks.

Dried preparations that contain lactose as the $\beta$-anhydride readily absorb moisture during storage and the β form then changes to the α-hydrate and the powder becomes caked.

If all moisture could be removed from dried byproducts there would probably be little change in the protein during storage. The solubility of casein and other proteins decreases with increasing temperatures of dry heating up to 307°F for 18 hours but there is no change in total nitrogen and little change in amide nitrogen.[82] Extensive degradation does not take place in dry casein until temperatures above 307°F are reached.

TABLE 17. *Changes in Dried Skim Milk of 7.3 percent Moisture after Storage in Nitrogen for 60 Days at 98.6°F.* (Data from Henry et al.[47])

| | | Fresh Powder | Stored Powder |
|---|---|---|---|
| Carbon dioxide produced (mgm./100 gm. powder) | | — | 5.8 |
| Apparent moisture content of the powder, % | | 7.3 | 6.7 |
| Equilibrium relative humidity, % | | 42 | 55 |
| Color of the powder, Lovibond yellow plus red units | | 0.5 | 2.2 |
| Solubility at 68°F, % | | 99 | 70 |
| Flavor of the reconstituted milk | | Palatable | Nauseating, caramelized gluey |
| Casein N | Amount present in the soluble portion of the powder, expressed as % of the total nitrogen in the fresh powder | 80.7 | 6.8 |
| Albumin N | | 2.3 | none |
| Globulin N | | 3.7 | 4.6 |
| Proteose and Peptone N | | 6.8 | 12.8 |
| Non-protein N | | 6.5 | 6.7 |
| pH of the reconstituted milk | | 6.73 | 6.50 |
| Reducing power,[8] moles x $10^{-5}$ ferricyanide reduced/gm. milk solids | | 0.9 | 16.0 |
| Free amino-*N* content of the protein, as % initial value | | 100 | 36 |
| Reducing sugar combined with protein, as mgm. lactose/gm. milk solids | | 6 | 49 |
| Biological value, method of Osborne et al. | | | |
| (gm. gain/gm. protein consumed) without added lysine | | 2.81 | 1.82 |
| (gm. gain/gm. protein consumed) with added lysine | | 2.59 | 2.55 |

Development of the stale flavor in powder was retarded by lowering its moisture content (Figure 10) but its appearance was not necessarily correlated with the temperature and time of storage.[77]

The season in which skim milk was produced was found to affect the keeping quality of powder made from it.[93] The initial palatability of dried skim milk produced from fall milk was higher than the powder produced from spring milk but the palatability of the fall milk decreased during 16 weeks storage at 80, 100 and 120°F. Similar powders from spring milk held at 80°F improved in quality during storage while those held at 100 and 120°F first improved, then deteriorated in quality. The seasonal differences were not observed in whole milk powders and it was concluded that the composition of the non-fat solids was responsible for the seasonal variation in keeping quality.

The gas packing of powders that contain no fat and which have less than 3 percent moisture does not appear to improve the storage stability of the products sufficiently to justify the added cost of removing the air from the container. Thiel and Pont[126] concluded that gas packing was not effective in preventing deterioration of dried skim milk.

Henry et al.[48] found no evidence that the rate of combination of protein amino groups and reducing sugar was directly influenced by the atmosphere in the container. However, they did show that dried skim milk remained palatable longer when air was removed from the container than when it was not. Table 18 shows some of their results. The nitrogen packed powder held its palatability longer in every case than did the air packed sample Recent work[26] on the non-lipid changes that occur in dried milk are in agreement with those cited above.

TABLE 18. *The Effect of Moisture Content of Dried Skim Milk upon the Number of Days Required to Render it Unpalatable.* (From Henry et al.[48])

| Storage temp. | | 3.0% Moisture | | 5.0% Moisture | | 7.6% Moisture | |
|---|---|---|---|---|---|---|---|
| °C | °F | Air-pack, days | *N*-pack, days | air-pack, days | *N*-pack, days | air-pack, days | *N*-pack, days |
| 20 | 68 | 500 | >700 | 410 | >700 | 115 | 340 |
| 28.5 | 83.3 | 370 | 650 | 290 | 590 | 12 | 51 |
| 37 | 98.6 | 240 | 450 | 145 | 260 | 2 | 18 |

The nutritive value of the proteins of dried skim milk slowly deteriorates during storage. Henry et al.[48] believe that loss in nutritive value stems in large measure from the inactivation of lysine. However, powders containing not more than 5 percent moisture were stored in air- or gas-packed containers at temperatures up to 83°F for at least 2 years without suffering a detectable loss in the nutritive value of their proteins.

Hodson and Krueger[51] found that dried skim milk which had become discolored and unpalatable during prolonged storage showed marked losses in arginine, histidine, lysine and methionine. With old samples which retained their color only minor and insignificant differences were observed.

## METHODS OF MANUFACTURE

The dried byproducts are manufactured by employing in succession the processes of forewarming, concentration and drying. These processes are modified in many ways depending upon the equipment at hand, the nature of the raw materials to be dried and the characteristics required of the finished product.

**Dried Skim Milk.** Dried skim milk is the most important of the dried byproducts of milk. The annual production of dried skim milk in the United States increased from 41 million pounds in 1920 to 481 million pounds in 1940 and 676 million pounds in 1947. The product is widely used in animal and human foods. It is an important ingredient in many special dried foods and in prepared dry mixes.

By an act of Congress in 1944 the terms "nonfat dry milk solids" and "defatted milk solids" were applied to dried skim milk for purposes of the Federal Pure Food, Drug and Cosmetic Act. This change permits food manufacturers to use terms other than dried skim milk on their labels.

*Drum dried skim milk* may be made according to the standard conditions used by Combs and Hubbard.[23] Preheat the milk to 150 to 160°F and pass it directly to the hot drums. Maintain the milk level over the drums at a point just below the bottom of the splash plates and even with the top of the drums. Use 60 pounds steam pressure and a drum speed of 24 r.p.m. Combs and Hubbard found the capacity of their 24 x 36-inch drums under these conditions was 58 to 60 pounds of powder per hour.

Specific directions for the operation of drum driers cannot be given because many variable factors must be considered. Drums are generally operated at a speed of about 16 r.p.m. Higher speeds than this sometimes cause objectionable vibration or chatter. It is customary to raise the temperature of the drums as their speed is increased. Practical relationships between the heat, drum speed and contact time is shown in Table 19.

The clearance between the two drums must be the same throughout the length of the drum in order that a milk film of uniform thickness will be deposited on the drum surfaces. The usual clearance between the rolls is about 0.020-inch.

The concentration of the skim milk to be drum dried may vary from normal to 40 percent total solids. Drier capacity may be increased by precondensing the skim milk in a vacuum pan to 18 to 30 percent solids. Many operators use 2:1 condensed skim milk for drum drying.

The output of a drum drier may also be increased by preheating the milk to as high as 200°F, and by raising the temperature and speed of the rolls. The effect of variations in these factors is shown in Table 20 compiled from results of Combs and Hubbard.[23]

The drum dried skim milk is scraped from the rolls in sheets, collected and coarsely ground before it is packed in bags or barrels.

*Spray dried skim milk* is made by atomizing skim milk in a stream of dry, heated air and then separating the dried particles from the moisture laden air. When the dried product is prepared for bakery use the skim milk is forewarmed to 180 to 190°F for 30 minutes. If the powder is to be used in ice cream or in other foods where a pronounced cooked flavor is objectionable the skim milk is forewarmed to 160 to 170°F. After forewarming the skim milk is concentrated in a vacuum pan or in the precondenser of a drying unit to 36 to 42 percent solids. The concentrate is fed to centrifugal atomizers or to high pressure sprays by means of a positive pump or homogenizer operating at 2,000 to 5,000 pounds per square inch pressure. The size of the liquid droplets varies from 5 to 125 microns.[92] The temperature of the skim milk at the time it is sprayed should be 150 to 185°F while the dried particles apparently are at 110 to 150°F.[92] The temperature of the air into which the milk is sprayed may vary from 200 to 300°F but an air temperature of 260°F is common. The temperature of the exhaust air varies from 130 to 190°F. The dried skim milk falls to the floor of the

TABLE 19. *Relationship between Applied Heat, Drum Speed and Time the Milk is in Contact with the Drum Surface.* (Data from three factories, Allen.[1])

| Heat Applied to Drum | | Drum Speed, r.p.m. | Contact Time, sec. |
|---|---|---|---|
| Steam Pressure, lbs./sq. in. | Interior Drum Temperature, °F | | |
| 30 | 275 | 10 | 4 |
| 45 | 293 | 15 | 3 |
| 100 | 338 | 20 | 2 |

TABLE 20. *The Effect of Skim Milk Treatment and Drum Adjustments on the Capacity of an Atmospheric Drum Drier. Size of Drums, 24 x 36 Inches.* (Data from Combs and Hubbard.[23]

| Milk Treatment | | Drum Adjustments | | Drying Capacity, lbs. / hr. |
|---|---|---|---|---|
| Concentration S.N.F., % | Temperature, °F | Speed, r.p.m. | Steam Pressure, lbs./sq.in. | |
| 9.2 | 55 | 24 | 60 | 53 |
| | 155 | 24 | 60 | 58 |
| | 185 | 24 | 60 | 69 |
| 9.2 | 55 | 36 | 85 | 64 |
| | 155 | 36 | 85 | 72.5 |
| | 185 | 36 | 85 | 85 |
| 30.0 | 40 | 34 | 85 | 70.2 |
| | 180 | 20 | 85 | 73.2 |
| | 180 | 28 | 85 | 100.0 |
| | 180 | 34 | 85 | 105.0 |
| | 180 | 36 | 85 | 130.0 |

drying chamber or is separated from the moist air by means of a series of cyclone collectors.

Dried skim milk is usually packed in 200-pound slack barrels fitted with heavy paper liners. Paraffin lined, tight oak barrels can be used for bulk export packing. The product will take up 1 percent moisture when held in these oak barrels for four months at 110°F and 80 percent relative humidity.[141] The compression of dried skim milk is discussed later in this chapter.

Spray dried mixtures containing half skim milk and half whey solids have been dried for special bakery use. Such a mixture may be forewarmed, condensed and dried in the same way as plain skim milk.

*Modified dried skim milk products* are prepared for use in manufactured foods or in special diets. Frequently, attempts are made to alter the condition of the milk protein to gain some advantage in body or water binding capacity. Sometimes mixtures of skim milk and other foods are dried together to produce a product that may be especially useful in food preparation.

The manufacture of a dried skim milk of high viscosity suitable for making a smooth textured ice cream is described by Mook.[85] Skim milk is forewarmed to 150°F, condensed to 40 percent solids, superheated by direct steam to about 200°F and held at this temperature until a slightly toughened coagulum is formed. The holding time may extend from a few seconds to 30 minutes, after which the curd and whey mixture is "smoothed out" and spray dried.

Sanna[109, 110, 111] coagulated the casein of skim milk at 195°F by the addition of hydrochloric acid to pH 4.4. The protein was redispersed with sodium hydroxide at pH 9.5 and then the reaction was finally adjusted to an optimum value between pH 7 to 9.5. The product was condensed and dried for use in ice cream or other foods. Lactose and salts could be removed from the product by draining part of the whey from the protein curd. The Sanna process is essentially one of producing sodium caseinate and mildly hydrolyzed protein from skim milk.

Dried skim milk has often been suggested as the basic ingredient in vaious kinds of desserts. Frequently, special procedures are used to manufacture the skim milk or the skim milk mixture. When dried skim milk is added to cream it improves its whipping properties.[103] Many reconstituted dried skim milks of 25 percent to 30 percent solids content can be mechanically whipped in a few minutes to a stiff white foam. The whip may be stabilized by rennet, acid or fruit juice[137] or by acid and potato starch.[45] Milk and citrus juice mixtures have been dried for dessert and beverage use.[6, 37, 88, 146] Dried milk-pectin mixtures can be reconstituted with water to produce jellies or beverages.[91,145] Dried milk-rennet mixtures contain the basic ingredients for easily prepared desserts[9, 84] and recently it was found that a lactone could be substituted for the rennet enzyme.[58] An apple-milk confection has been made by drying pulped apples and skim milk on a vacuum drum dryer.[133]

A special spray dried skim milk from which at least 15 percent of the soluble milk constituents were removed has been advocated for use in making soft types of cheese.[130]

**Dried Sweetened Milk.** Dried sweetened milk or dried sweetened condensed milk is a recently developed product, the varying composition of which is given in Table 21. The fat content may vary from fat-free to the full-fat or whole-milk Swiss product. The usual precautions observed in the manufacture and packaging of dried whole milk should be carried out in the preparation of a dried high-fat sweetened milk. The tendency toward development of an oxidized flavor during storage decreases as the fat content is lowered. A product such as Milk 4, Table 21 exhibits much better keeping quality than one of the composition of Milk 2. It has not been shown that sugar retards oxidation of the milk fat in those products in which fat is present.

TABLE 21. *Composition of Samples of Dried Sweetened Milk*

| No. | Dried Product | Fat, % | Milk-Solids-not-Fat, % | Sucrose, % | Moisture, % |
|---|---|---|---|---|---|
| 1 | Sweetened skim milk[148] | — | 39.0 | 59.0 | 2.0 |
| 2 | "Guigor"—a Swiss milk[14] | 16.73 | 46.01 | 33.60 | 3.66 |
| 3 | Sweetened, part skim[132] | 12.70 | 65.24 | 19.85 | 2.21 |
| 4 | Q.M.C. requirements[101] | 12.50 | 59.25 | 25.00 | 3.25 |

Sugar is used in the dried sweetened milks to improve their palatability and their redispersion characteristics. The presence of sugar does, however, limit the food uses of dried sweetened milk to those products which normally contain sugar. Dried sweetened milk is useful in the manufacture of chocolate coating, pie filling, cake frosting, and various types of dry milk preparations containing malt and cereal. It can be used in ice cream plants, in bakeries for bread and sweet goods and for chocolate milk drinks.

Manufacture of dried sweetened milk is begun by standardizing a fluid mixture of skim milk and whole milk or cream to the fat content needed to yield a dried product of the required composition. The milk is forewarmed to 190°F (range 180 to 200°) for 20 minutes. It is then drawn into the vacuum pan and concentrated to 36 to 42 percent solids. The required quantity of refined sugar is thoroughly dissolved in part of the standardized concentrate. The complete mixture is next warmed to 140°F and spray dried.

Mussett and Martin[86] described the experimental manufacture of a dried sweetened skim milk and its use in the manufacture of ice cream. A product of 14 to 20 percent solids was sprayed at 750 pounds per square inch pressure into a drier in which the incoming air was heated to 325°F. The flavor, color and texture of the finished powder was satisfactory for use in ice cream.

Dried sweetened milk is more easily produced by spray than by drum drying and the spray product is of higher quality. Rosecky[108] has developed a roll drying process with a disintegrator to be used in smashing the sticky ribbon of material delivered by the drums.

**Compressed Dried Milk Cakes.** Compressed milk cakes were developed during World War II as a means of saving shipping space and packaging material. Their value in a normal economy is questionable since some disadvantages developed. It was found necessary to break or grind the compressed cake into powder before it could be rehydrated. If moisture was absorbed during storage the cake became exceedingly hard and its solubility after crushing was reduced. Although compression was used as a means of reducing the oxygen content of packages of dried whole milk,[77] this did not greatly improve its keeping quality.[125] The density of spray dried skim milk as it comes from the drier is about 0.60. This value may be increased to 0.84, 0.89 and 0.97 by compression of the powder at 800, 1,600 and 3,200 pounds per square inch pressure, respectively.[141] At least 2,400 pounds pressure must be used to hold the powder in cake form. This is a higher pressure than can be attained with tile pressing equipment which was generally available during the war. In some cases binding agents such as crystalline dextrose, fat, glycerine or similar materials were employed to aid in caking the powder so that a firm block could be secured at pressures of less than 1,500 pounds. Hygroscopic powders are difficult to compress, especially on automatic presses, because they absorb moisture and become sticky. Dense, compact, dried skim milk is easier to compress than light, fluffy powder.

The density of a spray powder is affected by manufacturing conditions. Density as well as the size of the powder particle may be increased by increasing the solids content of the milk being sprayed,[78, 141] by severe preheating or superheating of the milk,[83, 85, 117] or by removing entrapped air from the milk before it is sprayed.[25]

The density of uncompressed roller dried skim milk varies from 0.3 to 0.5, depending upon the fineness of grind. The absolute density of air-free dried skim milk has been found to be 1.44.[77]

Special compressed food blocks that contained milk were prepared during the last war. Cakes of dried soups and cereal-milk-sugar mixtures were used for rations. Bars of milk flavored with chocolate and various fruits were made by compression for a school lunch program.[3] Cakes of dried milk that contained 20 per cent sugar and some vanillin were more friable, dissolved easier, and tasted and kept better than plain milk blocks.[125] Beverage and dessert blocks made from dried milk, sugar, cocoa or other flavoring and with 20 percent extra fat were molded or rolled into slabs. The fat formed the continuous phase and acted as a binder. No compression was needed. The ingredients were mixed with the melted fat and shaped in molds. For reconstitution the cake was placed in hot water, the fat melted and the milk solids were dispersed.[31]

Various coatings, such as one containing gelatin and a plasticising agent[62] have been advocated for covering milk blocks.

**Dried Buttermilk.** Dried buttermilk may be manufactured by either the drum or spray process. Sour cream buttermilk is usually dried by the drum process while that from sweet cream is customarily spray dried. There may be variations in processing and in utilization but generally the sour buttermilk is made for animal feed and the sweet buttermilk for human food. In 1947, 2.3 billion pounds of buttermilk was produced in the United States but only 0.5 billion pounds was dried. Approximately 0.8 billion pounds was not manufactured but was utilized without processing or discarded. McDowall has discussed the utilization of dried buttermilk in New Zealand[81] and Hunziker its manufacture in the United States.[60]

Sour cream buttermilk is high in lactic acid, or in lactates if it has been neutralized. These substances cause dried sour cream buttermilk to be more hygroscopic than the sweet cream product. Both dried buttermilks are more hygroscopic than dried skim milk.[30] The lactose in dried sour cream buttermilk should be crystallized to reduce its moisture absorbing properties, and to prevent the dried product from caking.

Collis[22] developed a single drum process for drying buttermilk which has now largely been displaced by the conventional double drums. Collis showed that severe agitation was necessary to maintain the insoluble curd particles in a finely dispersed state in the whey. Without proper mixing, the curd, which dries easily, and the whey, which forms a sticky, sirupy mass, reach the drums at different times, thus reducing operating efficiency.

Sour cream buttermilk should be heated to 160 to 170°F in a high-temperature short-time heater, then passed directly to the reservoir between the double rolls. Operation of the drums is the same as when skim milk is dried but the steam pressure should not exceed 80 pounds and the film should be cleanly scraped from the drums to minimize discoloration. Immediate packaging after drying will result in a caked product. Caking can be avoided where facilities permit, by allowing the lactose time to crystallize before the powder is finally packaged. This may be accomplished by taking the buttermilk from the drums at a moisture content of 5 to 10 percent. The powder is then held in barrels or bins for 24 hours before it is ground and sacked. Keeping quality will be improved by removing excess moisture after the lactose crystallizes. This may be done by subjecting the powder to a blast of hot air before or after grinding.

Sweet cream buttermilk is spray dried for human food by the same process that is used for skim milk. Dried sweet cream buttermilk should be tightly packaged to prevent moisture absorption. It should be stored in a cool place to retard discoloration and flavor deterioration. Dried sweet cream buttermilk can be used in ice cream,[127, 144] bakery goods, and in the manufacture of other foods.

**Malted Milk.** Malted milk, according to Federal Specifications[36] is "prepared under best sanitary conditions, by combining whole, fresh, clean milk with the liquid separated from a mash of ground barley malt and wheat, with or without the addition of sodium chloride, sodium bicarbonate, and potassium bicarbonate in such manner as to secure the full enzymic action of the malt extract, and by removing water. The finished product shall contain not less than 7½ percent butterfat and not more than 3½ percent of moisture." No definition or standard of identity for malted milk has yet been promulgated under the revised (June 25, 1938) Federal Food Drug and Cosmetic Act. Hunziker[61] has described the manufacture of malted milk in some detail. Horlick[56] has suggested the addition of pineapple or other fruits to the wort before it is dried. Pectin has been used to improve texture and increase viscosity.[145]

The composition of a typical malted milk is as follows:

| | |
|---|---|
| protein | 14.6% |
| lactose | 21.5 |
| fat | 8.5 |
| maltose | 1.5 |
| dextrins | 47.0 |
| ash | 3.6 |
| moisture | 3.3 |
| | 100.0% |

Since a milk fat percentage, but no milk-solids-not-fat percentage, is usually specified for malted milk, there can be a wide variation in the fat content of the milk used in the preparation of malted milk. A product with a composition shown above could be made with a milk of 2 percent fat. Malted milk can thus provide an outlet for surplus skim milk.

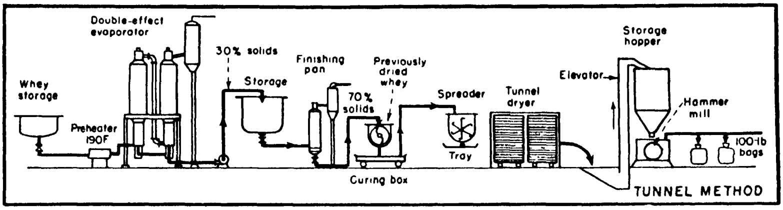
In the manufacture of malted milk a mash is made by mixing 100 to 250 pounds of ground barley malt with 100 pounds of cereal (generally wheat flour) and 4 or 5 times as much water. It is customary to steep the barley malt in water and to prepare the flour as a cooked paste before mixing them. The mash is then heated to 150°F and held for about 2 hours or it may be gradually raised to 170°F to more rapidly convert the starch to maltose and dextrin. The wort or liquid extract obtained from the mash is drawn off and mixed with sufficient milk to produce a dried product of 7.5 percent fat.

Salt is added to the wort-milk mixture and it is forewarmed and concentrated under vacuum in the usual way to a sirup of 60 to 70 percent solids. The malted milk sirup may be spray or drum dried but these operations are sometimes difficult because 70 percent of the solids of malted milk is lactose, maltose and dextrin. Drying is more easily accomplished in the conventional malted milk drying pans. These are special vacuum pans equipped with agitators and large doors through which the porous lumps of dried malted milk are removed.

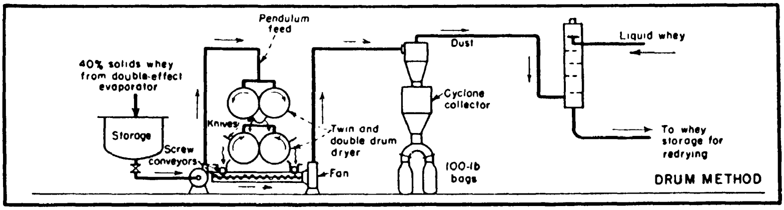
**Dried Whey.** Three general methods for the drying of whey have been developed since Bosworth[13] concentrated whey under vacuum and then dried it in air at 149°F. Whey is now dried by tunnel or shelf drying, drum or roller drying, and spray drying. Since many of the processes and their modifications employed in the drying of whey are patented, the status of the patents in this field should be investigated before manufacturing operations are started.

Flow sheets showing the important steps in the manufacture of the three types of dried whey as described by Stringer[121] are presented in Figure 11. It is common practice to concentrate the whey before subjecting it to one of the drying processes. Whey may be concentrated in individual batches in a single-effect evaporator or it may be continuously condensed in multiple unit systems. Two, three or four effects may be used to maintain an economical and efficient rate of water removal. A common practice is to concentrate fluid whey to about 40 percent solids in a triple-effect evaporator and to further reduce the water content to 60 to 70 percent solids in a batch type vacuum pan.

*Tunnel drying.* The commercial method of concentration and tunnel drying of whey is based on the Simmons patent[114] which only recently has expired. Whey is condensed under vacuum to 70 percent solids, dropped from the vacuum pan, seeded and held not more than 24 hours to allow the lactose to crystallize. If casein whey is used, the acid is neutralized by a lime slurry before drawing it from the pan.[121] Seeding may be done by the addition of a small amount of dried whey from a previous batch. The pasty mass is then spread on $\frac{1}{16}$- to $\frac{1}{8}$-inch mesh screen trays and dried in a tunnel drier through which air at 140°F is circulated. Figure 11 illustrates how the heavy concentrate may be handled mechanically. If drying air at 160 to 180°F is used, drying can be completed in 3 to 4 hours.[121] The dried whey after grinding is relatively non-hygroscopic and the whey protein is insoluble in water. In the Kraft modification of the Simmons process[68] the whey concentrate containing 70 percent solids is dried in air by blowing it through a series of conduits and cyclone collectors. The lactose crystallizes during this drying operation. Lavett[74] concentrates whey of 40 percent solids to 80 percent on a double drum drier, drops this to cooling and seeding drums, and then to a hot-air drier. Bertram and Lemmerich[12] pass the concentrated whey through a mixing machine in which it is mixed with air and the lactose crystallized. The material then is dried and ground. Peebles and Manning[98] claim that, by heat-coagulating the whey proteins prior to crystallizing the lactose in the concentrate, the protein then will not interfere with crystal formation, and a stable, non-hygroscopic dried whey will be produced.



**By concentrating, then either tunnel drying . . .**


40% solids whey from double-effect evaporator
Storage
Screw conveyors
Pendulum feed
Knives
Twin and double drum dryer
Fan
Dust
Cyclone collector
100-lb bags
Liquid whey
To whey storage for redrying
DRUM METHOD

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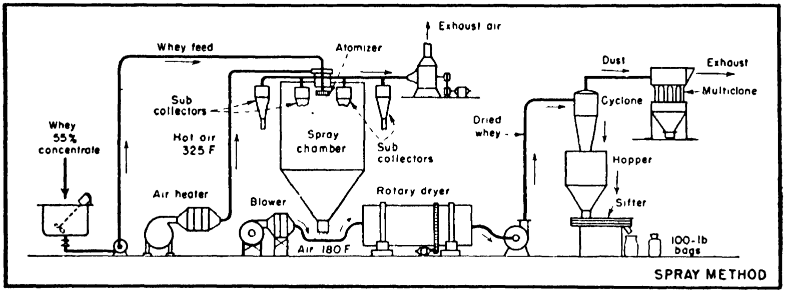

**. . . or spray drying—**

Fig. 11. Processes for drying whey. (*Courtesy of Food Industries and W. E. Stringer.*[121])

A whey bran feed for dairy cattle has been prepared and used in Sweden.[64] The product is produced by drying a mixture of 33 kilograms of rye bran and 1000 kilograms of fresh whey. The whey is concentrated to 15 to 20 percent moisture before it is mixed with the bran. The mixture is then tunnel or air dried.

*Drum or roller drying* of whey is reported to have been carried out by following conventional methods and without the use of drying aids or special processes.[4] The whey is condensed to 50 percent solids and the sirup is fed at the rate of 750 gallons per hour to a battery of drum driers. The rolls of each unit are $\frac{1}{64}$-inch apart and operate at 13 r.p.m. and at a temperature of 300°F. The whey dries in light brown crisp, broken sheets. These are ground and packaged.

For the successful operation of a drum drier it is important that the scraper knives be sharp and adjusted so they fit the drum surface closely. All whey must be removed during each revolution of the drum, otherwise it will gradually build up an insulating coating which will finally decrease drying capacity.

Modifications of the usual drum drying process generally are employed to retard the formation of sticky, hygroscopic glass on the drums. Lavett[71, 73, 74, 80] uses two double-drum drying units placed one above the other. A modification of this system is shown in Figure 11. The whey is concentrated under vacuum to 40 percent solids and then distributed throughout the length of the top rolls by an oscillating pipe. The upper drums reduce the whey to 80 percent solids. Twenty-seven pounds steam pressure is used on these drums and they turn at the rate of 2 r.p.m. The top drums revolve away from each other while the lower ones rotate inward. The concentrate drops in patches onto the lower drums where it is cooled to about 112°F in less than 60 seconds. The mass is seeded by the lactose crystals that adhere to the cooling drums and the crystallization proceeds during final drying in a rotary dryer at 130°F.

Both sets of double drums are used as drying rolls in another method.[121] Sixty pounds steam pressure is carried on the upper rolls and 35 pounds on the lower ones. The partially dried product is removed from the top rolls and dropped to the lower drums where it is finally dried.

Lavett[72] also developed a process whereby whey is concentrated to about 45 percent solids in a multiple-effect evaporator and then further condensed to 8 to 15 percent moisture on a double drum drier. The heavy layer is stripped from the drums and the lactose in it is allowed to crystallize as the $\alpha$-hydrate at a temperature below 199.4°F. Drying is continued by any suitable means.

When a single pair of rolls is employed, adjustment of the titratable acidity of the whey to between 0.30 and 0.40 percent before drying, is helpful.[75]

The use of drying aids such as skim milk, buttermilk, cereals and potatoes has been mentioned earlier in this chapter. When a feed or food use for the dried whey product is available, the addition of a drying aid provides a simple solution to the problem of drum drying whey. Spellacy[116] roller dried a mixture of 8 pounds of dried skim or buttermilk with 100 pounds of fluid whey. Satisfactory drying characteristics are usually obtained when the whey solids content of the mixture to be dried does not exceed 40 to 65 percent of the total solids of the mixture. A product useful in bread baking can be made by mixing fluid whey and skim milk in the proportions 35 percent whey and 65 percent skim milk, forewarming at 190°F, and condensing to 12 to 15 percent solids content, then drum drying the mixture.

*Spray drying* can be accomplished by the processes used for drying milk. However, the hygroscopic nature of spray-dried whey sometimes causes it to cake in the collection system of the drier and obstruct the passages. This tendency is lessened by inducing lactose crystallization before or during drying by an adaptation of one of the procedures previously described.

Stevens and Hufnagel[119] have dried whey on a 9-foot Grey-Jensen type spray drier by using the following procedure: Sweet rennet cheese whey was separated and forewarmed to 180°F for 20 minutes and condensed to 40 percent solids. The whey was concentrated in a single-effect vacuum pan because the drier was equipped with a dry collector which was not designed to precondense the liquid. The concentrated whey at 130°F was pumped at 2,000 pounds pressure to a 0.020-inch spray nozzle. The air coming into the dryer was heated to 285°F while the outgoing air was 225°F. The temperature of the powder at the collector was about 135°F. Difficulty with powder sticking in the collection system was avoided by the use of high drying and collecting temperatures. The lactose in the finished product was amorphous and hygroscopic in character but the whey protein was soluble.

Stringer[121] has described the commercial spray drying of whey on the equipment shown in the lower part of Figure 11. A method of atomizing the whey and a drying chamber design for use in this process has been patented by Peebles.[95] The whey is concentrated in multiple-effect evaporators to 50 to 55 percent solids. The lactose in the concentrate is crystallized under slow agitation during a holding period which may extend overnight. The condensed whey is pumped to a centrifugal atomizer in the top center of the drier. Here the whey is forced through the slotted openings of a disk whirling at 12,000 r.p.m. It emerges as a fine spray into a cylindrical drying chamber the size of which may vary from 15 to 30 feet in diameter. Air heated to 300 to 320°F is introduced into a plenum chamber above the atomizer and then down around the spray where it comes into intimate contact with the whey droplets. Rapid drying ensues and the particles swirl down through the cone shaped base of the drier into a duct where it is picked up by a current of air heated to 180°F and passed through a rotary drier. The whey discharged at the bottom of the cone usually contains 85 to 87 percent solids, whereas, the finished product from the rotary drier contains about 3 percent moisture. As the whey passes from the bottom of the drier cone through the rotary drier, the lactose crystallizes from its highly saturated solution. This requires one molecule of water per molecule of lactose. Excess water remaining after crystallization is removed in the rotary drier. The resulting product is granular, free flowing and nonhygroscopic in character but the whey proteins have been rendered insoluble during the process.

Modified dried whey products have been made for therapeutic use. Products consisting chiefly of the protein and mineral constituents of whey have been prepared by patented processes. One method is to precipitate the protein-mineral complex with alkali, separate it, wash or reprecipitate it to free it from any objectionable protein decomposition products, and finally dry it.[106, 135] Another method is to precipitate electrolytically, filter, and wash and dry the precipitate. A product thus is obtained containing approximately 18 percent calcium, 6 percent phosphorus and 20 percent protein.[20] Another therapeutic product consists of whey, an edestin-calcium solution derived from hempseed, and magnesium sulfate.[115]

**Dried Comminuted Shortening.** The current successful production and sale of prepared biscuit, cake and doughnut mixes was made possible through the development of dried shortenings. Some shortening preparations have been made by spray drying mixtures composed of a suitable fat dispersed in milk,[16] skim milk,[17, 35] whey[67] or any of these products including buttermilk.[90] When such a mixture is homogenized and dried the shortening globules become encased in envelopes of whey or skim milk. The milk byproduct coating remains water soluble so that the globules of fat are quickly dispersed when the prepared mix is converted to a dough or batter by the consumer.

A dried comminuted shortening may be manufactured by combining various procedures outlined in the references cited above. An edible hydrogenated or partially hydrogenated vegetable oil which is solid at room temperature and has good shortening properties is used. Sufficient fluid milk byproduct is added to the oil to reduce the fat content of the nonaqueous materials to 50 percent or more fat. One such mixture[35] contains the following components indicated in pounds: hydrogenated cottonseed oil 390, milk fat 10, skim milk 1,000, water 267. Another mixture[17] is a homogenized emulsion of cottonseed oil, 100 pounds; 1:1 concentrated skim milk, 500 pounds and; based on the dry weight, 0.2 percent soybean lecithin and 0.2 percent algin. Emulsifying agents such as a partial ester of glycerine and a higher fatty acid[90] or, as mentioned above, vegetable lecithin and an alginate,[17, 90] may be used. The mixture is heated to 160°F, homogenized at 2,500 pounds pressure and spray dried or in the case of the mixture containing lecithin and alginate, roller drying can be used.

**Dried Cream.** Spray dried cream and cream mixes are special products manufactured in relatively small quantities on special order for bakers, confectioners, and for use in aeration from patented gas cylinders. Before the cream is dried it is sometimes fortified with emulsifiers, stabilizers, antioxidants, sweetening agents, and additional skim milk solids. The addition of any materials not naturally present in cream removes the mixture from that class of products defined as cream under the Food and Drug Administration definition.

Dried cream usually contains about 72 percent fat and is made by drying a cream of 18 to 20 percent fat. High quality cream, quickly separated to 18 percent fat from fresh milk, is heated to 170°F for 20 minutes. It is sprayed at 2,000 to 3,000 pounds pressure into a dryer adjusted to furnish the minimum amount of heat needed to reduce the cream to 1 percent moisture. The powder is collected and cooled as soon as possible. Collection of the cream powder is the most difficult step in the drying process because the powder tends to stick to all contact surfaces. The collection system should be simplified to reduce the number of places, such as pipe elbows, where the hot dried cream can gather.

Slack barrels are satisfactory packages for dried cream that is to be held in cool dry storage for not more than 6 months. The product should be double gas packed in tins for ordinary commercial usage.

A dried cream product of improved miscibility, solubility, flavor and keeping quality has been prepared by dispersing lecithin in the fluid cream in an amount equal to 0.5 to 3 percent of the weight of the cream solids.[89] An increase in the solids-not-fat content of an 18 percent fat cream by the addition of an alkaline caseinate before drying has been advocated.[11]

Powdered cream mix to be used for whipping by aeration with nitrous oxide gas has been prepared.[100, 129] To cream of 19 to 34 percent fat is added 8 percent solids-not-fat, 5 percent by weight of sugar, 0.3 percent emulsifying agent and 0.1 percent vanilla concentrate. The mix is pasteurized at 160°F for 30 minutes, sprayed at a temperature of 150°F and a pump pressure of 200 to 1,500 pounds per square inch through a 0.0145 to 0.025 inch nozzle. The powder is cooled and gas packed in tins.

**Dried Ice Cream Mix.** Dried ice cream mix may be considered as a by-product of milk because it affords an outlet for surplus skim milk and it is dried on conventional drying equipment. Dried mix attained commercial importance during World War II and in 1947 approximately 14 million pounds was made. Ice cream mix is dried either for reconstitution and freezing in the usual commercial freezer, counter freezer or frozen custard machine, or for home whipping and freezing in a domestic refrigerator. The latter must have good whipping properties and sufficient stabilizer to prevent large ice crystals from forming during still freezing. Little information is available concerning packaged household mixes. The following process for manufacture of a commercial dried mix was assembled in part from information in several recent reports.[24, 99, 102, 143]

To manufacture dried ice cream mix, first combine the cream, milk and skim milk, forewarm them to at least 170°F for 20 minutes and condense them in a vacuum pan to 28 to 36 percent solids. Higher preheating temperatures, especially of the skim milk fraction, tend to retard the development of oxidized flavor during storage of the dry mix. The stabilizer and sugars may be added before or after condensing. Sucrose, dextrose and corn sirup solids are widely used but at least 75 percent of the total sugar should consist of sucrose.

Ten to 25 percent of the required sugar should be added to the mix before it is dried. The remaining sugar together with powdered vanilla, chocolate or other dry flavoring ingredients are blended into the dried mix. The purpose of adding only 10 to 25 percent of the sugar before drying is fourfold: first, mixes made without any sugar present during drying tend to churn in the freezer; second, drier capacity is increased when part of the sugar is withheld; third, when all the sugar is run through the drier there is a tendency toward the development of a caramel flavor; and fourth, the powder is less hygroscopic when part of the sugar is added to the dried mix in a crystalline state rather than when it is dried in an amorphous condition with the mix.

After the mix is condensed, the temperature is raised to 150°F and the mix is homogenized at 2,500 to 3,000 pounds per square inch pressure. The agitation during concentration and the effects of pressure spraying do not exert sufficient homogenizing action to produce a mix with optimum freezing properties. The homogenized mix is spray dried in the usual way so that the finished product will contain less than 3.0 percent moisture.

The dried mix should be continuously removed from the drier and immediately cooled to below 100°F.

Dried ice cream mix is blended with the proper quantity of sugar and gas packed in cans to maintain its keeping quality. The same problems of fat deterioration that arise during the storage of dried whole milk are also encountered with dried mix.[104]

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# 6

# CHEESE AND CHEESE PRODUCTS

The quantity of skim milk cheese made in the United States has increased from about 100 million pounds in 1932 to about 250 million pounds in 1947. A relatively small quantity of skim milk is used in the condensed or dried form in the making of cheese spreads. Small quantities of whey are utilized in making whey cheese and, as condensed and dried whey, in making cheese spreads. Buttermilk is not used by itself in making cheese or cheese products, but small proportions may be added to skim milk and whey utilized in cheese, as indicated later in this chapter.

## SKIM MILK CHEESE

The principal skim-milk cheeses made in the United States are the soft, uncured cottage, pot and baker's cheeses. Small quantities of Gammelost, Sap Sago and hand cheese are made in certain sections of this country. A small proportion of the total output of Cheddar cheese is made from skim milk, but, since the process used is a modification of making whole milk Cheddar cheese, it will not be discussed here.

**Cottage, Pot and Bakers' Cheese.**[4, 5] These cheeses are soft cheeses made from skim milk by practically the same processes, differing principally in the method of final treatment, and in methods of packaging and use. The skim milk is curdled by the action of acid developed in the milk by fermentation either with or without the presence of a small proportion of rennet to aid in the firming of the curd. Heat is the principal firming agent, but, if overused, causes the curd to become tough; rennet firms without toughening. Pot cheese is a relatively firm curd that has been washed, drained, and salted, and is packed in butter tubs or 5- or 10-pound cans for use in other food products. The same curd may be packed in 8-, 12-, or 16-ounce containers and sold at retail as cottage cheese. However, much of the present-day cottage cheese has had cream added and is known as creamed cottage cheese. Bakers' cheese is a soft curd that has been drained to the desired degree without washing, and may or may not be salted. It is marketed in tubs or cans and used in bakery products. These cheeses are highly perishable and will spoil rapidly unless refrigerated. If they are to be kept for a long time, they should be stored under brine in a refrigerator or should be frozen and kept in a frozen condition until used.

*Cottage cheese.* The equipment essential in the making of cottage cheese comprises a pasteurizer, a water-jacketed cheese vat with cover, a whey strainer, horizontal and vertical curd knives with wires spaced ½-inch apart, a rake for stirring, means for determining acidity, a floating thermometer, a 1-milliliter pipet graduated in tenths, a curd scoop, and butter tubes or straight-sided cans for holding curd. A mechanically-driven dough mixer is desirable for incorporating cream.

Times, temperatures, and proportions of starter given in the following directions are only approximate, mainly because of differences in the activity of starters. This should not be taken to signify that conditions of making should not be carefully controlled, but rather that they should be altered, usually within the ranges given, as may be necessary to produce a curd of the characteristics desired.

The freshly-separated skim milk should be pasteurized at 145°F for 30 minutes, or at 160°F for 15 seconds, transferred to the vat, cooled to 70 to 72°F, and from 1 to 3 percent of clean-flavored starter added, the quantity depending on the activity of the starter and the time that is to be allowed for incubation. The quantity suggested should be suitable for an incubation period of about 15 or 16 hours, or overnight. If the skim milk is available in the morning, and it is desired to complete the making of the cheese during the working day, the quantity of starter may be increased to 10 percent or even more, so as to shorten the incubation time to 3½ or 4 hours.

If the rennet type of cottage cheese is being made, from ¾ to 1¼ milliliters of rennet diluted with 50 milliliters of water are added per each 1000 pounds of skim milk. After adding the starter and rennet, the milk is stirred for a few minutes and then incubated at 70 to 72°F until a firm curd has formed and the acidity of the whey is between 0.50 and 0.55 percent. Higher temperatures may be used to shorten the time of incubation; in order to complete the incubation in as short a time as 4 hours, it may be necessary to incubate at temperatures as high as 86°F. It is claimed by some cheesemakers that a curd produced at these higher temperatures has less flavor than if produced at lower temperatures.

As soon as the whey is at the proper acidity, the curd is cut into half-inch cubes by first passing the horizontal knife lengthwise of the vat, then passing the vertical knife crosswise, and finally passing the vertical knife lengthwise. The temperature of the curd should then be increased at such a rate that it will reach a temperature in the range of 112 to 118°F in from 1 to 1½ hours after cutting. The curd should be stirred frequently enough to keep it from matting, but it should not be stirred more than needed and the stirring must be gentle, particularly while the temperature is below 100°F. Too much or too vigorous stirring breaks up the curd. If the vat is only partly filled with curd and whey, the first stages of heating may be accomplished by adding warm water to the curd with the temperature in the jacket slightly above 100°F. Water at a temperature of 110 to 120°F is run on the surface of the curd to the depth of 2 inches. After 15 minutes, water at 120 to 130°F is run in to raise the level another inch. Further heating is accomplished by increasing the temperature in the water jacket. This should ordinarily be not more than 15°F higher than the temperature of the curd. After the temperature of the curd has reached the desired cooking temperature, it should be maintained there until the curd is of the desired firmness. This is determined by putting a handful of curd in cold water and noting the degree of firmness after thorough cooling. Rennet type curd may be heated somewhat higher than 118°F and the time of heating required thereby decreased, but even for this type of curd the temperature should not exceed 130°F.

As soon as the curd is in the proper condition, the whey is drained off through a whey strainer and the water drained from the jacket of the vat. Water at about 75°F is then added in amount equal to that of the whey removed, the curd thoroughly stirred, and, after standing 10 minutes, the wash water is drained off. Two more washings are given with as cold water as is available. During the last draining, the curd is piled at the sides of the vat, a trench being left in the middle of the vat. After an hour's draining, the curd is ready for salting, or, if creamed cottage cheese is being made, for creaming and salting.

One to 1½ pounds of salt, according to taste, are added per 100 pounds of curd and stirred in. If cream is to be added, the salt should be dissolved in the cream, the salted cream added to the curd, and thoroughly mixed in. For a relatively dry product containing 5 percent fat, 12½ pounds of 40-percent cream should be added per 100 pounds of curd; for a moister product, 25 pounds of 20-percent cream per 100 pounds of curd. If a still moister cheese is preferred, the cream may be diluted with pasteurized milk before it is mixed with the cheese. The finished cheese is transferred from the mixer to butter tubs or straight-sided cans and chilled for at least 4 hours at a temperature below 40°F. It is then packaged and delivered for consumption within the next few days.

If it is necessary to store cottage cheese curd, it should not be creamed before storage, but should be covered with a brine containing 4 percent salt and refrigerated at a temperature below 40°F. The brine should cover the curd completely. Frequent change of brine will aid in preserving the flavor of the cheese. Preserved in this manner, cottage cheese curd may be kept in satisfactory condition for 2 to 3 months. When the curd is removed from storage, the brine is drained off, the curd is washed once with cold water and drained for an hour. It may then be creamed as directed above, or only salted. However, since the curd retains some of the salt of the brine, little or no salt may be required, and the amount to be added should be regulated by taste. Another method recommended for storing curd is to freeze it in paraffin-lined tubs or well-tinned cans and hold at about 10°F. The curd is drained, but not washed, before storing it by this method, and is washed, salted and creamed after removing from storage.

*Pot cheese.* Pot cheese is made by the procedures described for making cottage cheese. It is more often made without rennet, and the curd is cooked to a greater degree of firmness than is desired in cottage cheese. It may be salted, but is not creamed.

*Bakers' cheese.* In making bakers' cheese, the proportion of rennet may be as great as 10 milliliters per 1000 pounds of milk. The pasteurized skim milk is incubated at 72°F until the curd shows a clean break and the acidity of the whey is between 0.40 and 0.45 percent. The curd is then drained, usually in bags made of muslin. If a fairly dry cheese is desired, the bags of curd may be pressed, after whey has ceased to flow freely, by placing weights on them. The curd is not washed. It may be salted or not, according to the specification of the customer.

A small-scale procedure has been developed for making bakers' cheese from spray-dried skim milk.[10] This makes it possible for bakers to have fresh cheese available at all times and to avoid the costs of freezing, frozen storage and part of the transportation costs involved in marketing and preserving this cheese on a large scale. The relatively non-perishable dried skim milk can be stored at the bakery and converted to cheese as required. Practical bakery tests with cheese made by this method have given satisfactory results.

Eleven pounds of dried skim milk are added slowly to 89 pounds of water at 60°F with·constant high-speed stirring. The temperature of the reconstituted skim milk is brought to 86 to 90°F and 8 pounds of an active lactic-acid starter and 0.20 cc. of rennet are added. The milk is thoroughly mixed and allowed to stand for from 4 to 6 hours, during which time coagulation will take place and there will be a slight "wheying off." The acidity of a sample of whey from several inches below the surface should be from 0.52 to 0.58 percent (as lactic acid). The curd is dipped into muslin or fine-mesh flour bags and allowed to drain. The bags are piled several deep and repiled several times during the first hours of draining. After several hours the bags of curd are placed in a cold room at 35 to 40°F to complete the draining. This takes from 18 to 24 hours, after which the cheese is ready for use. An average yield of 22 pounds of cheese should be obtained.

**Gammelost.**[6] Gammelost is a cheese of 24 to 65 pounds in weight made from sour skim milk. The milk is heated in a kettle and allowed to stand for an hour to permit the curd to settle. The curd is gathered up in a cloth and put in a form in which it is lightly pressed. The cheese, still in the cloth, is immersed in hot whey for an hour, then removed and pressed again. It is transferred to a warm place and held there for 2 weeks, being turned daily, after which it is packed in a chest with wet straw around it to cure.

**Sap Sago cheese.**[6] This is a small, hard, green cheese flavored with the leaves of an aromatic clover, *Melilotus cerulea,* grown especially for the purpose. It is made from skim milk, to which buttermilk and whey are added in the making process. The hardness of the curd is regulated by the acidity of the skim milk used and the quantity of sour whey added. The suitable proportions of these factors are to be determined by experience.

The skim milk is allowed to develop some acidity, but not enough to cause it to coagulate on being heated. It is heated to boiling with constant stirring, cold buttermilk is then added, and this is followed by the addition of sufficient high-acid whey to cause a coagulum to appear on the surface. This coagulum is skimmed off and discarded, and then, with stirring, high-acid whey is added to precipitate the casein. Too little acid whey added at this point will cause the curd to be too soft, too much will cause it to be too hard. The curd is dipped and allowed to drain in thin layers until cool. It is then put in boxes and allowed to drain and ferment under pressure at 60°F for 3 to 6 weeks. The curd is then ground in a mill and 5 pounds of salt and 25 pounds of the dried clover added for each 100 pounds of curd. The mixture is worked into a dough and forced into molds lined with linen cloth. It is then packaged for marketing.

**Hand cheese.**[6] This is a small, low-fat cheese having a very sharp odor and taste. The product and the making process are different in different localities, but the most general procedure varies little from the following. Skim milk and buttermilk are mixed and held at about 70°F until the milk is sour and a thick curd has formed. The curd is broken up by stirring, heated to 120°F and held at this temperature for about 3 hours, the curd being stirred continuously during the first hour. The whey is drained off and the curd cooled. It is then ground, salted to taste, and pressed into forms. The cheeses are dried in a warm room and then transferred to shelves in a cellar for curing. As soon as the ripening has commenced on the surface, the cheeses are packed in boxes and the curing continued until they are considered ready for market.

## WHEY CHEESE

Because such a large proportion—approximately one-half—of the solids of milk used for making cheese is discarded in the whey, and because the lactose, whey protein and salts of whey are wholesome and nutritionally desirable as food, efforts have been made by cheesemakers to produce whey cheeses and to incorporate whey solids in other cheeses.

Whey protein can be coagulated by means of heat and used for making a cheese such as Ricotta, but since the proportion of whey protein to total whey solids is less than 6 to 100, the yield of cheese is relatively low. Very much greater yields can be obtained by evaporating the water from the whey and thus retaining all the solids as a paste or curd. This procedure, however, has encountered the difficulties arising from separation of the lactose in large, hard crystals that impart an objectionable coarse, gritty texture to the product. This problem has been avoided or solved in such ways as by keeping the proportion of whey solids in cheese mixtures small,[1] by removing the crystallized lactose from concentrated whey, by stirring concentrated whey during crystallization to keep the crystals small, by fermenting part of the lactose to lactic acid, or by hydrolyzing part of the lactose to glucose and galactose.[3]

The processes described below are presented only as typical ones; many variations of them are possible to adapt them to special conditions or tastes.

**Ricotta.**[6] This cheese, known also as Ziger cheese, contains principally whey protein, but may also contain as much as 15 percent fat, if no effort is made to remove fat from the whey. It may be made from a sweet whey, or from a whey made acid by inoculating with a starter and ripening or by adding sour whey. Three to five percent of buttermilk or skim milk is sometimes added to the whey to prevent the formation of fine curd, but the casein thus introduced alters the characteristics of the cheese and this feature is generally considered to be objectionable. The whey is heated nearly to the boiling point to coagulate the protein, which rises to the surface in a flocculent condition. It is skimmed off, salted to taste, and packaged. In a modification of the process, the curd is pressed in hoops for 24 hours and put for a time in a salt bath to which sweet cider or vinegar has been added.

**Mysost.**[6] Mysost, or primost, contains all the whey solids. It has a buttery consistency, a light brown color, and a mild sweet taste. Sweet whey is strained into a kettle and boiled with constant stirring over direct heat. The coagulated protein that rises to the surface is skimmed off from time to time. When the volume of the whey has been reduced to about one-fourth of the original, the protein previously skimmed off is returned to the kettle and thoroughly incorporated with the whey by vigorous stirring and the boiling continued. When the mass has become of the consistency of thin mortar, it is poured into troughs of wood or stainless steel and stirred with a wooden paddle until cool to prevent the formation of large crystals of lactose. When too thick to stir, it is packed into greased wooden molds, cubical in shape, and, after standing until thoroughly cooled, the cheese is cut with wires into blocks of retail size. The blocks are wrapped in tin foil, dipped into paraffin, and packaged.

## CHEESE SPREADS[7, 8, 9]

Cheese spreads are food products, usually of a buttery consistency, containing cheese and one or more other ingredients, such as milk or whey solids, emulsifying agents, relishes, seasonings, condiments and coloring matter. They differ from processed cheese and pasteurized cheese in that they are softer and may contain substances other than cheese and emulsifying agents. The processes of manufacture are similar, however, and hence cheese spreads may be considered, practically, though not legally, as modified processed cheeses.

The cheese spreads on the market are of three principal types: (a) cream cheese with pickles, pimentos, olives, etc.; (b) processed cheese of sufficient moisture content to be buttery in consistency; and (c) processed cheese to which concentrated whey or skim milk has been added, and which contains sufficient moisture and fat so that it spreads easily. Only the last of these three types is discussed here.

Cheddar, Swiss, Roquefort and Neufchatel are the varieties of cheese principally used in cheese spreads, but most other varieties may be used. Condensed and dried whey and skim milk are produced for use in cheese spreads by the methods given in Chapters 4 and 5, respectively.

Typical commercial cheese spreads contain 40 to 45 percent moisture, 25 to 30 percent fat, and 25 to 30 percent non-fat solids. The percentage of lactose varies between 2 and 7, the remainder of the non-fat solids consisting of salts, protein and other nitrogenous compounds.

A cheese spread with relatively high moisture and low fat percentages has an undesirable mealy texture; with increased percentage of fat, the consistency becomes more like that of butter. If percentages of both fat and moisture are high, the spread has a weak body and is unattractive in appearance. If the ratio of lactose to water is greater than about 16 to 100, which corresponds to about 6.5 percent of lactose in the spread, large crystals of lactose may separate, giving a gritty texture to the product. Principally because of the lactose present, cheese spreads are subject to the action of bacteria and molds. They should be pasteurized and should be packaged in hermetically sealed containers. The keeping quality of cheese spreads depends somewhat on their reaction; values in the range of pH 5.8 to 6.1 have been found most suitable. Citric acid may be used to increase the acidity of a spread, if this is necessary.

Dried or condensed whey imparts a weaker body to cheese spreads than does dried skim milk, but gives a distinctive sweet taste that is considered a desirable characteristic by many consumers. By the use of dried skim milk, a product can be made that not only will spread easily, but also may be sliced satisfactorily.

The cheese used should be a blend of old and young cheese. Old cheese imparts flavor, but, if used alone, is likely to cause the spread to be grainy and to leak fat. A spread made from young cheese alone lacks flavor and is likely to be too rubbery in texture to spread easily.

Sodium citrate, sodium potassium tartrate (Rochelle salts) or disodium phosphate may be used as emulsifying agent. The first is usually considered the most suitable; there may be a tendency to leakage of fat if disodium phosphate is used.

Typical cheese spreads may contain the following proportions of component materials per 100 pounds of product; 55 to 65 pounds of Cheddar cheese, 15 to 25 pounds of 40 percent cream, 5 to 10 pounds of dried whey or 5 to 15 pounds of dried skim milk, 2 pounds of sodium citrate, 0.5 pounds of salt, and water to make a total of 100 pounds. The proportions to be used should be calculated from the compositions of the materials available with the aim of making a product of predetermined composition. If condensed products are used instead of dried, it is obvious that allowance should be made for their water content. As mentioned above, cheeses of types other than Cheddar may be used.

For making cheese spreads, a steam-jacketed kettle designed for processing cheese or for making candy is suitable. The kettle should be provided with double-action agitators having a scraping member. Part of the cheese is added to the kettle, the steam turned on, and the agitation started. Some of the water is then added and followed by the emulsifying agent. The whey, or skim milk, is then added slowly, next the cream and the remainder of the cheese, and last the rest of the water. The temperature of the mixture is raised to between 160 and 180°F and maintained in that range for 10 minutes, water being added to replace that lost by evaporation. The amount of water to be added can be determined only by experience with the kettle used. While still hot, the semifluid mass is run into wooden boxes lined with tin foil and the ends of the foil folded over the exposed surface as soon as possible. The foil should cover the cheese spread completely in order to protect it from bacteria and molds. The boxes should be stacked and covered so that cooling is slow; slow cooling minimizes risk of spoilage.

If it is desired to incorporate a large proportion of whey solids in a cheese food, the following patented procedure may be followed.[3] Whey is pasteurized, cooled, and inoculated with a starter containing high-acid-producing organisms, such as the lactobacilli, which grow best at 100°F. The whey is incubated at the most favorable temperature until from one-fifth to one-third of the lactose has been converted to lactic acid, which will be when the whey contains from 1.0 to 1.7 percent acid. The fermented whey is then concentrated in a vacuum pan to one-seventh to one-tenth of its original volume and then heated to a temperature of 248 to 257°F under pressure until there remains only 10 to 20 percent of the lactose originally present in the whey. It is claimed that this heating hydrolyzes the lactose to glucose and galactose. All or part of the lactic acid is then neutralized by means of calcium carbonate, sodium carbonate, or disodium phosphate, and the pasty product processed with ground cheese in such proportions as to form from 20 to 40 percent of the final product.

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# 7

# BAKERY PRODUCTS

## INTRODUCTION

The baking industry is one of the largest consumers of concentrated and dried milk. Estimates of the production of the fluid, condensed and dried forms of milk and their utilization in bakery goods are presented in Table 22. An estimate[41] covering the past fifteen years of utilization of the solids of milk in commercial baking indicates that the annual use ranges between 1½ and 2 percent of this country's total annual farm milk output, or about a quarter of a billion pounds of milk solids.

The total consumption of milk solids for breadmaking increased regularly during the period before World War II, and in 1941 it is estimated that over 250 million pounds were used, with a yearly average of 246 million pounds for the period 1935–39. During the war years the supply of dried skim milk solids for domestic consumption was reduced, and the baking industry was regulated by War Food Order No. 1 in the amount of dried skim milk that could be included in bread formulas to 4 parts per 100 parts of flour (January 1943 to August 1944). These years cover the wartime period in which there was a larger commercial baking volume and somewhat restricted unit use of milk. When all types of milk products for baking were converted to a milk solids basis, the baking industry was found to have accounted for about 60 percent of the supply of milk solids processed for food purposes in 1943, indicating an increase even during restrictions.[34] During the middle of 1944 when the supplies of dried skim milk were considered adequate for all civilian requirements, the ceiling of 4 parts milk solids per 100 parts flour was removed, and now no restrictions are placed on the amount of milk solids that can be included in bread formulas.

The restrictions and economic conditions did not permit the prewar continuous trend toward a steadily increasing rate of milk use in baking. Although eventually the upward trend is expected to be re-established, observers recognize that economic pressures, reflected in higher costs of recent years, have caused a rather sharp decline in the volume of milk used in commercial baking. Prior to 1947, the use of milk and skim milk solids in the production of bread and rolls had been estimated at 3 percent of flour weight, although this percentage ranged widely between individual bakers. A recent estimate of the average milk solids content of white bread is about 1.5 percent.[41] The promotion of improvement of quality standards of baked goods is expected to reverse recent trends and turn them toward higher levels of milk consumption.

Since termination of the U. S. Department of Commerce bi-annual census reports of the baking industry in 1939, no official government statistical information has been generally available describing the industry's total consumption of the various ingredients of baked goods. Prior to World War II, various industry estimates[41] indicated that of commercial production, about one-third of the white bread and rolls contained dried milk or skim milk solids, about one-third contained various types of condensed milks, and the remaining third of the commercial bread and rolls contained milk solids from other sources, or no milk ingredient at all.

TABLE 22. *Production of Milk and Some Milk Products* and Their Utilization in the Baking Industry*

(Data based on estimates of production, Bureau of Agricultural Economics; estimates of baking utilization by American Dry Milk Institute and by a milk industry executive.)

| Milk product | Production 1947 | | Baking Industry | |
|---|---|---|---|---|
| | Product, million lbs. | Fluid equivalent, million lbs. | Product, million lbs. | Fluid equivalent, million lbs. |
| Fluid milks | | | | |
| Fluid whole | 119,300 | 119,300 | 11.0 | 11.00 |
| Fluid skim | 48,438 | 48,438 | 16.0 | 16.00 |
| Fluid buttermilk | 1,862 | 1,862 | — | — |
| Fluid whey | 12,293 | 12,293 | — | — |
| Concentrated milks | | | | |
| Plain condensed whole | 175 | 367 | 13.4 | 28.14 |
| Plain condensed skim | 402 | 1,206 | 110.0 | 330.00 |
| Sweetened condensed whole | 247 | 544 | 21.8 | 46.96 |
| Sweetened condensed skim | 543 | 1,629 | 183.0 | 549.00 |
| Evaporated whole | 3,208 | 6,737 | 6.0 | 12.60 |
| Dried milks | | | | |
| Dried whole | 165 | 1,319 | 5.5 | 44.00 |
| Dried skim (non-fat milk solids) | 678 | 7,701 | 217.0 | 2,213.00 |
| Dried buttermilk | 45 | 500 | 1.51 | 16.60 |
| Dried whey | 156 | 2,521 | — | — |

* Exclusive of cream and butter

Recently, a survey of the baking industry was attempted in order to determine why bakers include milk solids in their formulas, as well as other related information.[10] Only a 20 percent response to the questionnaires was obtained and the results must be considered with this fact in mind. In order of importance, the reasons for including milk of any kind in bread were found to be: (a) improvement in taste and flavor of the finished product; (b) improvement in nutritive quality; (c) improvement in inside and outside characteristics of the products; (d) improvement in keeping and toasting qualities; and finally, (e) increased yield per batch of dough.

## MILK BYPRODUCTS USED IN BAKING

The finest flavored bakery goods are made from whole milk products rather than from any of the byproducts of milk. This is because milk fat produces a characteristic pleasing flavor not duplicated by other known food constituents. Where economic conditions permit its use, bakery goods of unsurpassed quality can be produced by the use of whole milk or of its concentrates. However, the byproducts of milk are used to manufacture a large part of commercial bakery products. Even though the milk fat is partially or wholly absent from these byproducts they are highly regarded and widely used for the improvement in flavor and physical characteristics that they impart to the bakery goods.

**Composition of Byproducts.** The approximate compositions of concentrated forms of skim milk, buttermilk and whey that may be used for baking are shown in Table 23. Detailed composition figures and discussion of the fresh byproducts have been given in Chapter 1. For comparative purposes the compositions of concentrated forms of whole milk are also presented in Table 23.

The constituents of milk byproducts in which the baker is chiefly interested are the milk sugar and milk protein. Skim milk and buttermilk do not differ widely in the percentages of these constituents, but whey contains much less protein and more lactose than do the skim milks and buttermilks. For the baker, the important difference between skim milk and sweet cream buttermilk is that buttermilk contains a somewhat different protein fraction together with most of the lecithin of the cream from which the buttermilk was derived.

Special blends of sweetened condensed milk in which extra skim milk solids have been combined are prepared for use in bread. The composition of such a partially skimmed sweetened condensed milk is: fat, 6 percent; milk-solids-not-fat, 26 percent; sucrose, 42 percent; and milk-moisture, 26 percent.[41] This milk ingredient, used for making highly palatable bread, is a mixture of approximately equal quantities of whole and skim milk. The amounts of either may be increased or varied to meet the bread qualities desired.

**The Suitability of Byproducts for Bakery Use.** Opinion among bakers is divided as to the relative merits of the various byproducts and particularly the form of byproduct suited to manufacture of bakery goods. Skim milk is a byproduct of milk used extensively in baking. In the interest of food conservation it would seem desirable to increase the use of buttermilk and whey especially in those bakery goods which now do not contain milk ingredients.

TABLE 23. *Approximate Composition of Some Concentrated Milk Products and Byproducts Used in Food Manufacture*

| Product | Composition | | | | | | |
|---|---|---|---|---|---|---|---|
| | Sugars | | Protein, percent | Minerals, percent | Fat, percent | M.S.N.F., percent | Water, percent |
| | Lactose, percent | Sucrose, percent | | | | | |
| Milk | | | | | | | |
| Plain condensed | 14.8 | — | 9.3 | 1.9 | 10.0 | 26.0 | 70.0 |
| Sweetened condensed | 12.2 | 42.0 | 7.7 | 1.6 | 8.6 | 21.5 | 27.9 |
| Dried | 38.7 | — | 26.2 | 6.1 | 26.5 | 71.0 | 2.5 |
| Skim milk | | | | | | | |
| Plain condensed | 16.2 | — | 11.1 | 2.5 | 0.2 | 29.8 | 70.0 |
| Sweetened condensed | 16.2 | 42.0 | 11.1 | 2.5 | 0.2 | 29.8 | 28.0 |
| Dried | 52.1 | — | 35.7 | 8.0 | 1.0 | 95.8 | 3.2 |
| Sweet Cream Buttermilk | | | | | | | |
| Plain condensed | 15.0 | — | 11.2 | 2.4 | 1.4 | 28.6 | 70.0 |
| Sweetened condensed | 15.0 | 42.0 | 11.2 | 2.4 | 1.4 | 28.6 | 28.0 |
| Dried | 47.9 | — | 36.0 | 7.8 | 4.5 | 92.0 | 3.5 |
| Sweet Cheese Whey | | | | | | | |
| Plain condensed | 51.3 | — | 10.1 | 6.0 | 0.6 | 67.4 | 32.0 |
| Sweetened condensed | 28.7 | 38.0 | 5.6 | 3.4 | 0.3 | 37.7 | 24.0 |
| Dried | 72.7 | — | 14.2 | 8.5 | 0.6 | 95.4 | 4.0 |

*Dried byproducts.* This discussion will be confined largely to consideration of dried skim milk but the merits of using byproducts in dried form may be applied also to dried buttermilk and whey to a greater or less degree.

The specific advantages that make non-fat dried milk appeal to the baking industry may be listed:[8]

(a) It requires no refrigeration and will not freeze or sour.

(b) It is convenient to use because of freedom from moisture which permits accurate weighing, controlled acidity, and reduced losses during handling.

(c) It is uniform in quality when obtained from a reliable manufacturer.

(d) Its use promotes certain economic advantages such as improved water absorption.

A recent survey[10] indicated that the amount of non-fat dried milk used in bakery foods was determined by increased consumer acceptance, availability and cost.

There is a difference of opinion as to the relative desirability of the two types of dried milk for bakery use. Many bakers prefer roller-dried skim milk because in their opinion larger volume, and closer grain and texture are obtained; the objectionable feature that an increased cooked flavor accompanied use of roller-dried skim milk is found in some instances. A difference in the baking behavior of the two kinds of dried skim milk is generally noted when experimental comparisons are made, but the varied conditions under which each type is produced by different manufacturers prevents a positive statement that one type is superior to the other. Differences in concentration of milk at time of roller drying or spraying are thought to be related to baking quality of the dried product, and the extent of preliminary heat treatment has definitely been shown to be a factor. Usually a high water absorption is correlated with good baking properties, and a convenient test is to mix equal amounts (10 grams) of powdered milk and water, and to note the type of the resulting mixture. Spray-dried milk, with its more soluble casein, produces a thin slurry, while the roller-dried product thickens to a consistency of paste. This test generally can be relied upon to distinguish the two types of dried skim milk, but it does not completely define water-binding ability over a period of time, nor guarantee baking behavior. Different characteristics of the dough are apparent when roller-dried skim milk is compared with the spray-dried product. The former, with its high solubility index (low solubility), tends to produce stiffness in the dough during the first part of mixing which hinders thorough mixing of dough ingredients. As mixing continues, the dough becomes progressively softer until it becomes difficult to handle. The spray-dried skim milk tends to form a soft dough from the start of mixing. A blend of the two has been found more satisfactory during mixing than either type by itself; usually a mixture of 30 parts of roller with 70 parts of spray has been found to give best results.[30]

Further evidence of differences between roller- and spray-dried process skim milks is provided by x-ray studies. X-ray analysis indicates that the different methods of manufacture cause the proteins of skim milk to show different unit spacing of refractant groups; spray-process dried skim milk shows slightly higher spacing than the roller-process powders.[40] The significance of this difference with respect to baking quality has not been explained.

In order to determine the baking quality of dried skim milk without carrying through the prolonged and sometimes indecisive experimental baking test a modified farinograph procedure has met with some success.[19]

The absorption of a mixture of equal parts of flour and milk solids is determined after 10 to 14 minutes mixing in the Brabender farinograph. When the absorption of the flour is known, the absorption due to the milk solids can be computed. This represents a combination of the physical absorption of water by the milk solids and the actual softening action on the gluten. Skim milk solids of good baking quality give absorption values of 70 percent or greater while the poor baking quality material gives values of 65 percent or lower. The method does not approach baking conditions, but has the advantage of showing the effect of the milk solids on the gluten development and on absorption. Bakers sometimes use dried skim milk to improve the crust and toasting color of bread, this characteristic being derived from the lactose of milk.

*Condensed byproducts.* The sweetened condensed milks are a smooth, homogeneous mixture of milk constituents and sugar, which blend quickly and easily with the other ingredients of bakery goods. They are preferred by many bakers as a source of milk solids. This preference is founded on the belief that better flavor, superior mixing properties and greater smoothness can be obtained with the sweetened condensed milks than with other forms of milk solids. Fluid forms of milk, especially blends of milk and sugar, are considered to excel in retaining milk flavor during processing and in transferring this flavor to the bakery product in which the milk is used.

Evaporated whole milk, packed and sterilized in 8-pound hermetically sealed tins, may be used to advantage in small or moderate scale baking operations, for bread, rolls and sweet goods.

Plain and sweetened condensed skim milk are the concentrated milks used most extensively by bakers (Table 22). The whole milk concentrates produce the best flavor in bakery foods, but they are more expensive than fat-free byproducts. A partially skimmed sweetened condensed milk containing over half as much fat as the whole milk product has been favored by many bakers. This blend of whole and skim milk with sugar has long been successfully used to manufacture high quality bread and cake at moderate cost to the baker.

Excessive age thickening occurs in inferior sweetened condensed milks under usual storage conditions and in all products during high temperature storage for long periods of time. Some of these relationships have been discussed in Chapter 4. Excessive thickening complicates flowing, mixing and removal from the barrel. Thickening is often accompanied by the development of off-flavors in the milk.

Plain condensed skim milk is often used by bakers who are located near a source of constant supply. Freshly prepared plain condensed milk shares the flavor transfer and smooth blending characteristics of the sweetened product but it has the disadvantage of being highly perishable. It must be held under refrigeration and stocks should not be accumulated or stored for more than three weeks.

*Frozen milk* appears undesirable for baking purposes. The denaturation of the casein and the changes in the other components resulting from freezing over any considerable length of time are not the same as those that occur during heating of milk prior to drying.

*Miscellaneous products* available to the baking industry are basically dried skim milks which have been modified by special procedures designed to produce various improvements in dough characteristics, flavor modifications or fermentation alterations when the product is added to the dough mixture. An increase in the lactic acid content of the dairy ingredient is frequently desired and may be accomplished by addition of lactic acid itself or by introduction of various cultures of lactic acid-producing bacteria. The cultures are inactivated after the desired acidity has developed and the final product is obtained after special drying and powdering operations.[16, 23] Acid producing and peptonizing bacteria are used also in producing various dairy ingredients for bakers.[5] These byproducts are principally isolated fractions of skim milk possessing such characteristics as improved whipping properties or high protein content.

**Processing Byproducts for Bakery Use.** A discussion of the processes used for the manufacture of the condensed and dried byproducts has been presented in Chapters 4 and 5. Byproducts to be used for bakery goods are manufactured following the usual procedures except that a special heat treatment of the milk has been found to be necessary in order to obtain optimum baking results.

## EFFECT OF THE HEAT TREATMENT OF MILK ON ITS BAKING QUALITY

A definite and rather severe heat treatment prior to concentrating and drying is necessary if the resulting powder is to have satisfactory baking properties. Controlled experiments indicate that soluble whey protein in a milk product, either dry or condensed, is undesirable in baking. Heat treatment coagulates the whey proteins and thus avoids the harmful effects of the soluble protein. The physical dispersion, degree of denaturation, and particle size of the casein are probably factors contributing to the baking properties of the various forms of milk. The casein of roller-dried skim milk is insoluble and that of frozen skim milk may also become insoluble but the differences in physical state of the two caseins is such that only the former has good baking quality. There appears to be a relationship between the amount of soluble protein in a milk (determined as nitrogen), and the baking quality of that milk. But the correlation between baking quality and soluble protein has not been 100 percent, and it has not replaced the customary experimental baking test that is recognized as the best method for determining baking quality of milk products. The physical state of the whey protein in skim milk seems to exert more influence on baking properties than the condition of the casein. However, a complete explanation with experimental data is still lacking.[20]

Greenbank et al.[13] were the first to publish data showing that the baking properties of skim milk were improved by heating it to temperatures above 170°F. Maximum bread volumes were obtained within the range 185 to 203°F. Further studies were made at temperatures from 145 to 212°F. for 30 minutes, in which bread baking results were obtained on loaf volume, fermentation tolerance, grain, texture and break.[14] Greater improvement in the bread was noted with soft wheat flours than with hard wheat flours, these tests being run with skim milk heated to at least 163°F. No explanation was offered for the improvement in baking quality brought about by the heat treatment of the skim milk.[13, 15]

Additional studies over a wide temperature range have been made.[33] The method of heat treatment, the length of time for the heat treatment, and the bread formula all introduce variables such that duplicate comparisons are difficult to obtain even if all other conditions are identical. With a standard straight dough formula (low fat) in which all the liquid was skim milk which had been given various temperature treatments in closed containers for twenty minutes, data were obtained which are plotted in Figure 12. The increase in volume of the loaf with increase in the temperature of treatment of the milk is evident. Similar increases are obtained with other types of heat treatment. Above 210°F this method and time of heating cause discoloration of the skim milk which carries through to the finished loaf, causing poor crumb color and cooked flavor in the bread. However, the volume increase resulting from heating the milk above 210°F is less significant than the volume increase from heating in the range 140 to 200°F, which is considered necessary if the skim milk or the powder made from it is to have satisfactory baking quality.

The changes that occur during the heat treatment of milk seem to center around the sulfhydryl radical of the whey protein. Sulfides are liberated when milk products are heated and the decrease has been measured quantitatively.[35] It was found that the heat labile sulfides originated chiefly from the whey proteins, in those products where the fat content was low.[39] Farinogram measurements have indicated that the dough softening factor of unheated milk is present in the whey portion, and that a similar dough softening action could be obtained if cysteine hydrochloride was added to the dough. The quantitative determination of the amount of sulfhydryl groups in raw milk indicated there was a sufficient amount of this radical to cause the dough softening action.[37] Polarographic analysis of raw milk produces a curve that had the cysteine-cystine characteristics, but after suitable heat treatment of the milk, the same curve can not be obtained.[37] In each instance the whey fraction, and probably the albumin portion, with its accompanying sulfhydryl groups appears to be involved. After heat treatment the sulfhydryl radical and the dough softening factor are greatly diminished.

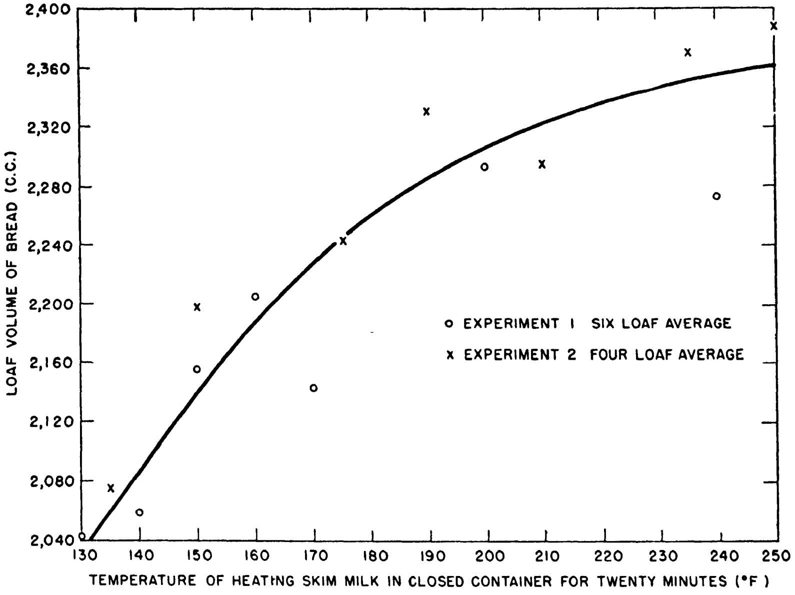

Fig. 12. The effect of heating fluid skim milk in No. 3 cans for 20 minutes at different temperatures upon the volume of 1-pound loaves of bread made to contain 6 grams of skim milk solids per 100 grams of flour. A time of 5 to 20 minutes was allowed to bring the temperature of the milk to and from the heating temperatures of 130 to 250°F.[32]

However, a more detailed examination of the amount, distribution and changes of the sulfur content of skim milk before and after heat treatment uncovered the facts that; (a) the amount of volatile sulfides lost during the heat processing was too small to account for the disappearance of sulfhydryl groups on heating, and (b) the removal by conversion of single sulfhydryl groups to double sulfhydryl groups (cysteine conversion to cystine) was found not to occur since the cystine content of whey protein did not change after heat denaturation, and cystine could not be detected in either the natural or denatured protein.[24, 25] An explanation was resorted to in which rearrangement and aggregation of the protein mycelle was believed to occur during heat treatment. This enclosed the sulfhydryl groups and made them inaccessible to direct titration, thus accounting for an apparent decrease in this group. This decrease in active sulfhydryl groups, following heat treatment, was thought to be responsible for the improvement in the baking properties of the skim milk.

In direct contrast to the results just mentioned, others have found that there is an increase in the reducing groups following heat treatment of milk.[17] This apparent contradiction may be explained by considering that as one reducing system is destroyed by temperatures above 158°F, another is liberated. The quantitative determination of sulfhydryl groups by the polarographic method is apparently very specific for this one reducing group, while determination of reducing groups by the thiamine disulfide method measures the reducing power of several groups. Since different methods were used in each case, this may also account for the contradictory results on the effect of heat treatment on the reducing groups of milk. The time and temperature of heat treatment as well as the amount of contaminating copper present seem to affect the reducing groups that can be quantitatively determined. They also add to the variables involved in comparing different sets of data. Apparently time of storage does not appear as a variable. It is interesting to note that if the thiamine disulfide reagent is used for measuring reducing groups in good and poor baking quality dried skim milk, no pronounced differences are found for the two types of powder.

## SKIM MILK IN BAKERY GOODS

**Skim Milk in Bread.** Precisely what happens to skim milk solids in flour doughs during baking has not been fully explained owing to the complexity of the system. Some observations have been made of the effect of dried skim milk on flour proteins, particularly with respect to gluten degradation.[4] The addition of skim milk to flour in amounts equivalent to 6 percent dried skim milk in the dough showed that the flour gluten was reduced in particle size (degraded) as indicated by failure to be precipitated with magnesium sulfate from solutions of the gluten in sodium salicylate solution. The difference in amount of gluten degradation by good and poor baking quality skim milk was not significant however, and apparently the effect does not help explain differences in baking quality.

In contrast, skim milk also appears to act as a strengthening agent to flour proteins, although a direct combination of skim milk solids with wheat gluten proteins has not been established. Very little conclusive evidence has been accumulated relating to the supplementary action of skim milk solids on flour proteins, but it is known that the increase in loaf volume brought about by including skim milk in dough is much more pronounced with medium strength flours than with strong flours. One of the more positive reasons for the use of milk is found in the improved characteristics of bread made from medium strength flours supplemented with skim milk.

Although the explanation of the beneficial effects of potassium bromate additions to the baking strength of certain flours is the subject of considerable debate, evidence is accumulating to indicate that its effect is centered on the flour proteins and not on flour enzymes. It has been observed that 6 percent dried skim milk reduces the damage from excess amounts of bromate, and creates a tolerance toward bromate additions in those flours which have already been brought to their optimum oxidation condition through other oxidizing agents prior to bromate addition.[28] This poising effect of skim milk solids may be due to the bromate acting on them, rather than the gluten, or to the fact that the gluten has been modified by the skim milk in such a way that bromate additions are not able to act in their usual manner.

While the whey proteins are considered responsible for undesirable effects on gluten properties, the casein is related to proper absorption, and 100 to 125 percent absorption by dried skim milk results only after satisfactory heat treatment. It appears that heat treatment is necessary to effect beneficial changes in both the whey proteins and the casein. Probably the changes in these proteins are independent of each other.

*Effects of skim milk on the bread making process.* The presence of skim milk is important in its effect upon absorption, mixing time and fermentation of the bread dough. The ordinary absorption range of 58 to 64 percent for bread flour can be increased when skim milk solids are included in the formula since the water absorption of the milk solids is normally much higher—in the range of 125 percent by weight. A general rule of thumb is that for each pound of skim milk solids an additional pound of water can be incorporated in the dough (or 100 percent absorption by weight).

High-speed mixing, contrasted with the slow-speed mixing of some years ago produce improved grain, texture, color and eating quality of the bread; increased absorption is brought about permitting increased bread yield, providing variables in temperature, formulas and bread-making procedures are controlled. When the optimum mixing time for water doughs is compared with that for milk doughs, no difference is observed, and generally a 10-minute mixing time is considered optimum for commercial doughs. However, mixing tolerance for milk doughs is found to be superior, and definite advantages are found with milk doughs over water doughs when both are subjected to excessive or over-mixing (e.g., 14-minute mixing time). Overmixed milk doughs recover much more rapidly and show little evidence of over-mixing by the time the dough reaches the pan stage; crust color of such milk doughs is normal, and excessive shrinking is not observed upon cooling of the bread. The explanation for the increased mixing tolerance of milk doughs is not known definitely, but measurements of dough plasticity during mixing indicate that milk doughs reach maximum plasticity at about the same time as water doughs. Milk doughs, however, reach a higher maximum plasticity which is maintained for a longer period of time.

Although individual steps in the process of bread fermentation are fairly well understood, the entire process is most complex and the order and relationship between the several stages is variously interpreted. One definite change that occurs during fermentation is an increase in acidity to a degree dependent on length of fermentation. Milk doughs usually show slower and less extensive increase in acidity as indicated by hydrogen-ion concentration changes; this may be explained by the resistance to change of acidity, or buffering action of the milk solids. By their influence in retarding acidity changes, milk solids may be said to influence indirectly other enzymic as well as physical-chemical changes occurring during fermentation.

At a level of 6 percent skim milk solids, a lower acidity is always obtained in comparison with water doughs, which is maintained throughout the fermentation period; the magnitude of the acidity change is considerably less at the end of fermentation for milk doughs, although both types of doughs become more acid as fermentation proceeds.

The changes in acidity of dough effected by including milk solids probably alter the gluten properties, but a more direct influence is found on the enzyme complexes present in dough. Milk solids seem to have a depressing effect on proteolytic and diastatic enzymes, and with flours of low diastatic activity the addition of milk solids may be unsatisfactory unless enzyme action is stimulated. The effect of milk solids on enzyme activity seems to be centered in the reduced acidity, since the optimum pH for diastases is usually considered to be in the range of 4.7, while average milk doughs show a pH of 5.5, and water doughs an average of 5.1. An increase in acidity to offset the effect of milk may be sufficient to stimulate enzyme activity. An increase in diastatic activity by addition of diastatic malt in conjunction with milk solids causes very marked improvement in the baking quality of flours of low diastatic activity that otherwise might be unsatisfactory when used with dried milk. The improvement seems even more pronounced with winter wheat flours of moderate strength. Excessive supplementing of milk doughs with diastatic malt may bring about increased activity of proteolytic enzymes and excessive browning of the crust, both of which are undesirable.

One method for obtaining proper diastatic activity during fermentation when milk is included is to add an amount of malted cereal flour to milk before drying, such that the cereal product does not amount to more than 1¼ percent of the non-fat milk solids. This process insures a product that permits sufficient diastase activity during fermentation without separate addition of diastatic supplements. Such a product cannot be sold as "dried milk" and is usually marketed under special trade names.

Milk solids have a direct effect on yeast activity since they decrease the amount of sugar available for yeast growth by reducing the activity of the starch converting diastases, largely through the repressed acidity changes. This reduced acidity in itself is more or less undesirable for optimum yeast activity, although yeast does show a wide range of pH for optimum activity. Milk solids influence yeast activity in a more obscure way when used in conjunction with yeast foods. Milk solids greatly improve the efficiency of yeast foods, especially when used with moderate strength winter wheat flours. The explanation for advantages secured by use of yeast foods in milk doughs is not available, but it is known that the fermentation rates of milk and water doughs are equal when a fermentation period up to five hours is used. Milk solids are important as a source of available nitrogen in the latter stages of bread fermentation, which may have a bearing on the observed results.

A combination of the influences of skim milk on the factors involved in fermentation of bread dough is best reflected in its effect on fermentation time. An extended fermentation tolerance is an advantage in bread production, and ample evidence is at hand to show that use of 5 percent milk solids, included in either sponge or straight dough formulas, acts as a dough stabilizer, and permits a 10 to 30 percent increase in fermentation time during which satisfactory bread can be produced. It is essential that sufficient fermentation time be given to milk dough, and care should be taken not to take the doughs on the "young" side.

*Effect of skim milk on the physical characteristics of bread.* The addition of milk or skim milk to dough produces definite physical improvements in the finished loaf making it more acceptable from the consumer's point of view. The earliest investigators noted that skim milk imparted a more desirable golden-brown crust color to the finished loaf than to loaves made from water dough. It was originally assumed that the milk sugar was responsible for this improvement. Subsequent investigations, however, show that no single component of the non-fat constituents of milk is responsible, and that only when all are supplied in the form of whole or skim milk is the desired crust color obtained. It is thus impossible to state precisely whether the lactose, casein, or whey protein has a greater effect. Apparently all contribute in some degree.

The same factors that contribute to improved crust color are also responsible for the improved toasting quality of milk bread. When milk solids are included in bread to be used for toasting, a safety factor in the rapid production of uniformly colored toast is guaranteed.

The crumb color as well as the texture and grain of the finished bread is equally improved by addition of skim milk to the formula. Soft, more velvety texture with complete absence of the harshness of water bread, and a grain of small uniform cells characterize the crumb of milk bread. The improved crumb color may be attributed in part to the improved texture, since small uniform cells appear lighter than large cells. The improvements in texture and grain as well as general eating qualities are also closely connected with the stabilizing influence of skim milk on fermentation. The improved grain and texture are reflected also in the improved slicing quality of milk bread. The moist, uniform crumb, and firm non-flaking crust of milk bread are ideal for non-crumbling, non-clogging, rapid slicing, and the increased moisture is essential in preventing excessive drying of the sliced loaf.

With the use of skim milk, a characteristic break and shred are obtained and there is a decided tendency to keep the break from running wild. A poor crust color not only results frequently with water doughs, but buckled bottoms and shrunken sides of the finished loaf are frequent. Milk doughs properly handled avoid all these undesirable characteristics and produce loaves of excellent symmetry and characteristic golden-brown shred. The effect of milk solids on volume depends on the method of handling the dough, the type of flour, and the characteristics one desires to produce in the finished loaf. With improper handling of milk doughs a reduced volume may easily result, but under proper conditions an equal or larger loaf volume can be obtained than with water doughs. Laboratory experiments consistently report 5 to 10 percent increase in loaf volume through the use of milk, while commercial production indicates that milk solids produce only a slight increase in volume; the greatest improvements are found in grain and texture. Commercial production often aims at producing an extreme balloon type of loaf, the volume of which cannot be further increased by skim milk. With the balloon type loaf, milk solids permit the desired volume to be obtained without greatly sacrificing the grain and texture of more compact loaves.

*Effects of skim milk on the keeping quality of bread* have been the subject of numerous investigations, but definite conclusions have not been reached. Bread staling is commonly thought to be the result of loss of moisture from the loaf, but it has been found that bread stales rapidly in sealed containers from which no moisture can escape. Moisture content is involved in staling, but it is not the only factor. The softness and moisture content of bread are not necessarily related to staling. At present, most of the softening agents added to bread have an effect on softness rather than on the actual process of staling. It is doubtful if skim milk retards or increases staling itself, but it definitely increases the moisture content of the loaf and allows it to remain soft for longer periods of time than similar loaves made without skim milk. By virtue of the moisture retaining ability of skim milk, the moisture of the loaf is increased and the loss of moisture from the bread is slower. Milk solids quite probably play a part in the moisture equilibria between gelatinization of starch and hydration of gluten that are established during and subsequent to baking. However skim milk has not been shown to be connected with the process of starch retrogression and aggregation, the principal processes of staling.

A great deal of conclusive evidence is at hand to establish the fact that skim milk in bread does not increase or decrease the rate of mold development in stored bread. Contamination of bread after baking, prior to wrapping, is the main cause for development of mold growth. The humdity at the surface of the bread, and the greater concentration of nutrients in milk bread are relatively insignificant factors. Much work has been carried out comparing the susceptibility of milk and water breads to mold growth under a variety of baking, cooling and storage conditions, with the conclusion that susceptibility is about equal for the two types. Plant sanitation and protection during cooling are the most important factors in reducing mold development.

*Effects of skim milk on the nutritional value of bread.* From the long range point of view, probably the most significant benefit resulting from addition of any type of milk solids to bread is the improved nutritional value imparted to the bread.[11, 32] Water bread is known to be rich in carbohydrates and low in minerals, proteins and vitamins. It is relatively simple by the use of any form of milk to increase the mineral content of bread, to improve its protein quality, and to supplement its carbohydrates. It should be pointed out that skim milks in the forms commonly employed in bread making contain substantially all the proteins, minerals, milk sugar, and water-soluble vitamins of milk. With the inclusion of skim milk in bread, the nutritional deficiencies of bread can not only be completely overcome, but the bread can become a chief source of dietary essentials. The 51 percent milk sugar of skim milk solids does not cloy the appetite, and, since it is not destroyed during fermentation, it is available as a nutrient in the finished loaf.

The chief protein complexes present in milk are, first, casein—the phosphorus-containing constituent present in amounts approximately equal to 75 percent of the total protein present, and second, the whey protein component, which although low in phosphorus, contains an abundance of sulfur and comprises the remainder of the total protein. Milk proteins are made up of as many as 20 amino acids, and since they correspond closely to the amino acids present in animal tissue, milk protein is considered of exceptionally high quality. Skim milk added to bread greatly increases its lysine and tryptophane which are deficient in cereal proteins, and in addition to this supplementing effect, there is an increased utilization of the cereal proteins. Thus, the biological value of milk bread is higher than could be expected from either of the protein components alone.

Skim milk improves the mineral composition of bread just as it does its protein composition. Calcium, phosphorus, and the potassium are present in high percentages in skim milk, and of these calcium is by far the most significant. Insufficiency of calcium in the diet is one of the chief nutritional problems, and the addition of milk solids to bread greatly increases the intake of readily available calcium.

*Effects of skim milk on the production cost of bread.* Although cost is one of the governing factors in the use of milk solids in bread, a careful consideration should be given to the many improvements effected by addition of milk solids to dough contrasted with their cost, and a thorough investigation of the cost situation should be made before conclusions are reached. The increase in the quality of the bread is probably more significant than the increase in cost. The actual cost per pound of skim milk solids cannot be be used, as a rule, for measuring the amount of skim milk solids to add since the influence on dough yield, fermentation and baking yield are such as actually to effect a saving on the total cost per pound of bread. The greatest saving accompanying use of skim milk is easily recognized when it is remembered that for each pound of skim milk solids an equal amount of water can be incorporated in the dough, and the yield of dough is increased by a like amount. Actual reduction in cost per pound of finished bread depends chiefly on current economic conditions, but data assembled from commercial firms show that with solids of milk and skim milk in the formula a reduced cost per pound may be obtained. A further saving is found with a reduction in returns of old bread, since milk bread retains its added moisture and accompanying softness longer than water bread.

*Effects of skim milk in specialty bread.* The inclusion of skim milk in a wide variety of rye bread formulas offers the same advantages as in white bread formulas. Usually the skim milk solids are added to rye doughs at the dough stage, but in place of adding an equal amount of water, addition of only half as much water as dried skim milk has proven most satisfactory. Using the straight dough procedure, the amount of water may be increased somewhat. Excessive water in rye dough causes spreading or flattening of the loaf during proofing and baking, and should be avoided. A few trial runs will indicate the proper amount of water to add under shop conditions. Skim milk retards the development of acidity, which on first thought might be considered undesirable for the development of the desired acid flavor in rye doughs. However, the desirable flavor-odor of rye bread is brought about by the production of volatile compounds easily detected by taste or smell. Their production is not always directly proportional to the development of acidity. It is thought that these flavor constituents are related to the type of fermentation occurring in the rye dough. The inclusion of skim milk in rye sours permits increased fermentation which is more effective in producing the flavor constituents than is the reduced acidity in permitting their development. The grain, texture, color, flavor, slicing properties, and symmetry of a loaf of rye bread are all improved by skim milk to a point which more than justifies its inclusion in the formula.

For the production of exceptionally nutritious, high quality bread, up to 12 percent skim milk solids may be included in the dough formula. With this level it is especially important to use milk solids of guaranteed baking quality, as well as to observe certain other precautions, since the addition of this amount of a milk ingredient will alter dough-handling properties considerably. The general rule of increasing water absorption by an amount equal to the amount of dried skim milk is valid at the 12 percent level, but the dough should not come from the mixer in a slack condition. Over-mixing should be avoided and the dough should be as stiff or slightly stiffer than doughs containing 6 percent milk solids. It is also most important that the sponge temperature at doughing-up time should not be in excess of 82°F, since higher temperatures will favor development of such undesirable qualities as excessive loaf volume, poor crumb color, inferior texture and flavor. Usually a sponge set at from 70 to 72°F with a sponge time of five hours will produce a temperature at dough time of 82°F. If the properties which are characteristic of a "young" dough are apparent, resulting from the reduced sponge temperature, a somewhat extended mixing time (12 to 14 minutes) and sponge time (6 hours) will usually correct the difficulty. Dough temperature does not seem as critical and an average temperature of 78 to 80°F with usual floor time is satisfactory.

Frequently, a high percentage of sponge produces undesirable weakened loaf body in high level milk bread and care should be taken that the sponge percentage does not exceed 60 percent (55 percent sponge has produced excellent bread of this type). Additions of diastatic malt may be necessary because of the reduced enzyme activity caused by skim milk solids, and it is usually desirable to add the malt diastase in the dough stage to prevent excessive slackness of the sponge. The sugar content of high level milk bread should be lowered to approximately 3 percent for best results during proof and baking.

Excessive moisture during proofing should be avoided, and slightly reduced proofing time is desirable since this type of formula shows larger oven spring than is usual. Baking temperature may be slightly reduced, and oven time slightly increased, but extreme changes are unnecessary.

It has been the experience of Australian workers that an increase of shortening to a level of 20 percent was necessary to produce satisfactory bread at the level of 12½ percent skim milk solids, but it must be kept in mind that the bread formulas were different from those used in this country and the typical Australian wheat produces flour of different baking quality from that of bread flour generally used in the United States.

**Skim Milk in Sweet Goods.** Sweet goods in the baking industry is a general term used to include a variety of products with the general requirement that the sugar content be 16 percent or greater. These products may be subdivided into classifications of yeast-raised and chemically-raised sweet goods; the former is illustrated by various forms of "roll-in" sweet dough (Danish dough), or plain sweet dough, and the latter by cakes and cookies. Doughnut dough may be raised by either type of leavening agent. A great variety of formulas and procedures are used in the production of sweet goods, depending on the particular requirements of the consumer.

Up to the present time there has been a gradual decline in home production of bread in favor of bread produced by commercial concerns. In the field of sweet goods, home production is significant and the baking industry is constantly alert for methods that will enable it to equal the quality of the home baked products. This has been done most effectively through the use of increased amounts of quality ingredients, including milk products, which produce improved color, flavor, texture and keeping quality in cakes, cookies, and other sweet goods.[2] The danger of using even slightly sour fluid milk is of course absent when dried or sweetened condensed milks are used and when these are employed for making plain baked or creamed custards there is less tendency for curdling or liquefying. When milk solids are used in making icings, especially chocolate icings, the excessive sweetness is reduced and the milk-chocolate flavor is accentuated. Icings containing milk solids hold up unusually well.

Since differences in formulas are sometimes considerable it is difficult to state precisely the exact content of milk solids contained in various types of sweet goods. However, within reasonable limits the following may be considered typical; figures represent parts of skim milk solids per 100 parts of flour used in the formula.

| | | | |
|---|---|---|---|
| Yellow layer cake<br>White layer cake | 10–12 | Pound cake | 5–10 |
| Devil's food cake<br>Chocolate cake | 5–8 | Plain sweet rolls<br>Danish rolls | 2–6 |
| Angel food cake<br>Sponge cake | 0 | Doughnuts | 5–6 |
| | | Cookies | 3–10 |

Any of the forms of milk (fluid, concentrated or dried) are used in cake production, depending on convenience and economy. Milk is considered a minor ingredient by some since it rarely accounts for more than 3 percent of the total cost of the cake ingredients and occasionally it can be replaced by water without undue harm. In commercial pound cake production where vegetable shortening sometimes replaces part of the butter content of the formula, fluid milk is used to advantage in balancing the water, since vegetable shortening is practically water-free.[6] In some cake formulas it is possible to replace part of the egg with skim milk solids, but identical results are not obtained. Frequently, in small scale cake production the general practice is to use fluid milk as the liquid component of the cake batter. In this instance, the effect of too little or too much liquid in the batter should not be connected with properties of milk, but to lack of proper balance of flour, sugar and liquid in the formula.

*Types of skim milk products used in cake making.* Uniformity of ingredients is essential for commercial cake production, and even more important for experimental baking. For the standard cake baking test as prescribed by the American Association of Cereal Chemists dried skim milk reconstituted in water was recognized as far more uniform than fluid milk that might be available in various laboratories.[7] However, several grades and types of dried milk are also available, and some attention has been given to studying the effect of these types of milk solids on cake production. The roller-dried skim milk showed increased absorption and greater emulsifying effect than the spray or vacuum drum dried products. These improvements carried through to the finished cake and produced greater volume, better symmetry, and resulted in higher scores in comparative cake judging. In spite of these advantages, however, and because of the greater solubility and uniformity of the spray-dried product, it was recommended that spray-dried milk be used in standard cake baking formulas and procedures. Specific reasons for the observed differences were not determined, although it is suggested that formalin titration of the reconstituted milk from each type of dried product might assist in selection of dried milk of proper physical condition.[7]

Other forms of whole or skim milk can be used in cake formulas with equal advantages. Corrections in the formula are usually necessary to convert the milk product to the equivalent of fluid skim milk, and in the case of sweetened condensed milk products an allowance for the sugar must also be made.

*Effects of skim milk on cake quality.* The addition of skim milk solids to cake formulas produces effects similar to those produced in bread and they will not be discussed here. These are listed, without reference to the order of their importance:[3]

(a) Moisture content of cake is retained for long periods of time.

(b) Appearance of interior and exterior of cake is improved; golden-brown crust is produced with complete absence of oiliness or greasiness.

(c) Flavor, richness, and taste-appeal are improved.

(d) Nutritional value is increased by addition of vitamins, minerals and proteins.

*Effects of skim milk on hydrogen-ion concentration of cake.* The importance of hydrogen-ion concentration on crumb color and flavor of cake has led to investigation of the effect of skim milk on the pH value of cake.[36] When dry skim milk was used at levels of 8, 15, 20, 25, and 30 percent with variations in water to maintain constant batter viscosity, a decrease in pH value over a range of 0.41 pH units was obtained. This indicated that under the conditions used, with a yellow-cake formula, there was a slight increase in acidity toward neutrality of the batter, with increasing percentage of milk solids. Commercial spray-dried skim milk was used in these experiments with a relatively slight variation in pH values of the cake between 7.12 and 7.41. All of the cakes scored high and were of good quality. The conclusions reached from this work were that dried skim milk affected the pH of cake to a limited and insignificant degree when used in amounts varying between 8 and 30 parts per 100 parts of flour.

**Dried Skim Milk in Prepared Mixes.** The use of skim milk in baked goods promises to be greatly expanded with the recent development of "prepared mixes" for the housewife and the commercial baker. It has been estimated that over 200 firms are manufacturing prepared mixes on a commercial scale to be used in producing a wide variety of bakery goods including cakes, cookies, doughnuts, pie crust and bread, as well as various types of yeast-raised sweet goods. Further estimates state that over 400 million pounds of wheat flour per year are used for pre-mix preparation, about half of which will be used in family mixes, and the rest for restaurants and bakeries. Advantages in convenience, economy and uniformity of resulting product seem well established.

The extent of increasing popularity of prepared flour mixes is indicated by the results of the survey undertaken by Cook in 1948,[10] which has been mentioned in connection with general usage of milk in bread. Under the conditions of his survey, 38 percent of the individual bakery proprietors use some form of prepared flour mix; two-thirds of this number use the mix for doughnut products. Cake pre-mixes were found to be of second importance with chocolate flavored types predominating, although spice, gold and white cake mixes were also used. The use of prepared mixes for sweet-dough products was significant, with 20 percent of the bakers reporting pre-mixes were used for all sweet doughs.

One factor that has stimulated the commercial development of prepared mixes for bakery goods has been the production of non-greasy, free-flowing, powdered shortenings which are highly resistant to development of rancidity and are able to withstand conditions of long storage on the grocer's shelf.[9, 12, 22, 23, 27] The development of satisfactory dried yeasts has also contributed to the success of prepared mixes of the yeast-raised types. Improved leavening agents coated with materials permitting long storage without losing potency have also been important.

The improved prepared-mix shortenings are of several types, one of which seems to have been specially developed to resist rancidity, while others consist of normal fats to which have been added antioxidants. Another type especially developed for prepared mixes is made by coating the fat with the solids of skim milk, buttermilk or whey. The coatings dissolve when placed in contact with water or when heated above 110°F. This coated shortening has an additional advantage in that it reconstitutes easily to produce a smooth batter. It also increases the absorption of the shortening. The milk solids become thoroughly dispersed throughout the baked product and increase moisture retention. The proportion of milk solids and shortening may be varied in order to provide the proper amount of each ingredient in the baked product; 35 percent shortening and 65 percent skim milk solids has proved satisfactory. The use of milk by-products in the manufacture of dried shortening is discussed in Chapter 5.

**Skim Milk in Cake Icings.** In addition to the use of skim milk in regular cake icings, advantages are obtained when it is included as a constituent of vegetable butter coatings and fillings for sandwich cookies. The improvements in the physical appearance and the ease of handling during manufacture of icings and fillings are indicated by the increase in the demand for the dried product for this purpose.[26] Other concentrated forms of skim milk including plain and sweetened condensed milk can be used with equal advantage, providing allowance is made for the water or sugar content.

The proportion of fat and dry ingredients used in vegetable butter summer coatings should be in the range of 32 percent fat, and 68 percent dry materials. For making winter coatings, 35 percent fat and 65 percent dry materials are used. The dried milks are of course included in the dry materials and from 15 to 20 percent is the amount generally recommended. The proper ratio of fat to dry materials must always be maintained and when dried skim milk is included in a coating formula the other dry materials (usually the sugar) must be reduced by a corresponding amount.

The inclusion of skim milk in coatings and fillings is definitely associated with elimination of the undesirable greasy texture sometimes observed when skim milk is not used. The flavor of a filling becomes rich and delicate when it is prepared with milk, especially when milk fat is present. Milk solids offer a further advantage in reducing excessive sweetness of fillings, thus preventing cloying of the appetite and inducing increased consumption. The problem of lessening the sweetness of the thicker type fillings is best accomplished by including amounts up to 25 percent of skim milk solids in the formula, thus producing a more pleasing product.

## WHEY PRODUCTS IN BAKERY GOODS

It has been recognized for some time that whey is a very acceptable food product. Difficulties in handling and processing related to the inherent composition of whey have delayed widespread acceptance of whey as a food product, but definite progress has been made, and the prospects for extensive use of whey products in the future are promising.

Although the undesirable factors in milk that may be responsible for poor baking quality seem to be centered in the whey fraction, considerable effort has been directed toward producing a whey product satisfactory for baking purposes. The effect of heat treatment on the chemical changes in the whey fraction has been studied.[39] Various economic considerations have been responsible for the interest in this problem. Commercial applications have been made,[29] but published data have not been extensive, and confirmation of results is lacking in some instances.

**Whey Products as Vitamin Enrichment Ingredients.** Some of the early efforts toward utilization of whey in baking[1] were designed and carried out with the objective of using the riboflavin of whey to supply enrichment levels of this vitamin. A number of bread baking tests usng straight dough baking procedure and dough ingredients generally used by commercial bakers were carried out. In these, dried whey and other whey products were investigated in experimental baking to determine their value as a source of riboflavin and also as bread ingredients compared with skim milk solids. Some of the dried whey materials were especially prepared for use in bread dough and showed wide ranges in riboflavin content. The bread formulas included the dried whey products at 4 and 10 percent (based on flour) levels using both high and low protein flours. The bread produced was satisfactory in all respects. In general, the wheys with riboflavin potency of 260 to 580 micrograms per gram were more acceptable and there was no indication that the dried whey produced grey crumb color, or that the bread doughs handled differently from the control. When used at levels of 4 percent whey, and 3 percent shortening, the dried whey was equal to standard non-fat milk solids in producing bread, except that the doughs with whey were softer and did not take as high absorption (3 percent less).

**Types of Whey Products Used in Baking.** *Fluid, concentrated and dried wheys.* For experimental purposes, good baking characteristics can be imparted to whey by proper heat treatment, and the product may then be prepared in liquid form, in various stages of concentration, or in the dried form. For the most part the baking results are equivalent regardless of the form of whey used. The type of flour used in conjunction with the whey introduces variations in the baking results. When used with low protein flour at the 10 percent level, dried sweet rennet whey produces satisfactory bread; with high protein flour, dried wheys at the 4 percent level produce dough of satisfactory handling qualities, while acid cheese wheys are poor in this respect. Neutralized casein whey is generally satisfactory, but Cheddar whey should not develop excessive acidity since accompanying flavors may carry over into the bread. Cottage cheese whey is less desirable for baking purposes, since it is apt to carry undesirable flavors and require special processing.

*Modified whey products.* The most extensive investigations on utilization of whey in baking have recognized that plain dried whey does not give the loaf volume that a baker desires, that additions of dried whey soften the dough to some extent, that additional mixing time is required, and that water absorption of whey doughs is altered. To meet these objections, a modified whey product was developed in which these undesirable effects of plain whey were reduced or eliminated.[32] A product of satisfactory baking properties was produced by adding skim milk and calcium salts to the liquid whey prior to processing. Calcium may be added as the sulfate, lactate, or phosphate or as the corresponding acid, which is subsequently neutralized with lime. Various proportions of skim milk may be used but a blend of equal quantities of skim milk and whey has been found to give excellent results in both bread and cake. The skim milk-whey-salt mixture is heated to develop good baking properties in the milk proteins, after which it is spray dried.

This modified whey product has good nutritional qualities as shown by rat growth studies.[1] Acceptability of bread made with the product is said to be equal to that of standard bread. By means of the increased milk solids and added calcium salts, the protein and ash content are increased and the lactose content is decreased in comparison with that of the unmodified whey. An approximate analysis of the modified whey product shows: moisture 4.5 percent; acidity 0.08 percent (on a reconstituted basis pH 6.5); protein 16.1 percent; lactose 57 percent; fat 0.9 percent; ash 14.1 percent; calcium 2.78 percent; and phosphorus 0.87 per cent.

It is thought that the improved baking quality of the modified whey product is brought about in part by the complete coagulation of the whey protein and possibly the partial coagulation of casein caused by the addition of the calcium salts and the subsequent heat treatment. The chief function of the calcium apparently lies in its effectiveness as a protein denaturing agent, especially in conjunction with heat and acidity. High temperature heating for an extended time will also improve the baking properties of whey but the improvement is not as complete and not as conveniently accomplished as when calcium is used.

**Dried Whey in White Bread.** During World War II, the U. S. Army Quartermaster Corps found that dried whey would be suitable as replacement for dried skim milk in producing white pan bread by the straight dough procedure under experimental baking conditions. However, the supply was inadequate for extended use by the armed forces. The dried whey used in these investigations was a commercial product from cheese sources, and was used in the bread formula at a level of 6 percent, producing very satisfactory bread as evidenced by results from the Quartermaster Food Acceptance Laboratory (400 people) when compared with standard army bread. Crumb characteristics of the whey bread were normal; flavor, texture and aroma could be distinguished only by direct comparison. The machining characteristics of the whey dough indicated no difficulty.

Several English workers have investigated the effect of adding different levels of commercial dried whey on the physical and nutritive properties of white bread.[18] A roller-dried whey was used in the experiments and it was found that up to 5 percent of the bread flour could be replaced with the whey without deterioration of the bread crumb, but a decreased loaf volume was obtained with some flours and a cheesy flavor was imparted to the bread at the 5 percent level. The calcium content, true digestibility of the bread, the "protein value," and the vitamins of the B complex were found to be improved at all levels of whey addition over control bread made without dried whey.

A more detailed study of the effect of commercial powdered whey on the biochemical processes involved in making bread was carried out by J. Svenson.[38] He found that the addition of powdered whey in amount equivalent to 3 percent of the weight of the flour slightly retarded the rate of gas development, but no change was noted in the total amount of gas produced when compared with control doughs made without whey powder. Improved moisture retention, bread yield and bread texture accompanied the addition of whey powder to bread formulas, but there also was some decrease in total fermentation time, when comparisons were made with control formulas. The addition of whey powder brought about a desirable hardness in the crust of rolls, as well as excellent crust color in both rolls and bread.

## BUTTERMILK IN BAKERY GOODS

Because of the lecithin and unique protein fractions in buttermilk, this byproduct should have advantageous baking properties, particularly in the dried form, and the small amount of published experimental evidence indicates that this is true. Most attention has been directed toward the use of buttermilk in various types of bread although it is believed that it can also be used to advantage in cake formulas.

**Buttermilk in Bread.** Dry sweet-cream buttermilk solids have been included in white pan bread formulas at the 6-percent level, and when compared with dried skim milk solids, at the same level a larger loaf volume was obtained.[31] It is believed that the lecithin content of the buttermilk solids is responsible for the increased loaf volume, since the addition of a small amount of soybean lecithin emulsified in butter oil to skim milk before drying, produced a product yielding the same loaf volume as the buttermilk powder. The addition of butter oil alone to skim milk did not effect the same result. Buttermilk with titratable acidity as high as 0.1 percent can be processed into powder of good baking quality providing it is neutralized with sodium hydroxide or lime to the normal acidity of buttermilk.[31] In the manufacture of canned bread it is desirable to maintain a comparatively high acidity, and, when cultured buttermilk solids are used in the formula in place of skim milk solids, a somewhat lower pH of the crumb can be obtained.

The use of buttermilk in rye bread has indicated definite advantages both on the experimental and commercial scale.[21] Buttermilk as a flavoring agent in rye bread has been used for many years, but the variation in acidity of the buttermilk often caused undesirable variations in the rye dough acidity and has somewhat limited the use of this milk byproduct. Controlled manufacturing techniques can easily assume a uniform product for baking purposes. Dried buttermilk of 11 percent acidity has been used commercially to prepare a mixture of buttermilk solids and medium rye flour in a proportion of 15 to 85. This is said to insure a uniform acidity in the rye bread dough. Buttermilk is responsible for marked improvements in flavor, crust color, crumb texture and structure, and keeping quality in rye bread. Less crumbling during slicing, increased water absorption, and improved nutritive properties and toasting quality are additional advantages.[21]

## FORMULAS AND PROCEDURES

Suggestions for typical formulas of bakery goods containing milk byproducts and for precise schedules for their production can be made only with the understanding that some variation will be necessary in order to meet local requirements. More rigid control of formulas and schedules is necessary in experimental investigations than in commercial production where day to day differences are always the rule, and where no two bakeries may be able to follow identical procedures. It is usually necessary to make several trial runs with the supplies and equipment at hand, and to make adjustments for ease of operation and quality of product on the basis of the results obtained. The following formulas are intended to illustrate some uses of milk byproducts in baking. They should be modified to suit individual needs. For formulas and methods of production of "specialty" bakery products commercial formula books should be consulted.

The milk byproduct specified in the formulas is "skim milk solids." This may be added in the form of plain or sweetened condensed skim milk by adjusting the quantities of materials used depending upon the milk solids and sugar content of the byproduct. Table 23 should be consulted for composition figures. If skim milk is used in dried form, the formula value for "skim milk solids" indicates the weight of powder to use. Frequently, it will be desirable to reconstitute dried milk with some of the water in the formula before the ingredients are finally mixed. Fluid and condensed milks may be used without preliminary treatment. Sweetened condensed milk is normally a homogeneous blend or milk ingredients and sugar in a form ready for bakery use.

Various combinations of whey, buttermilk and skim milk solids may be substituted in the formulas for the "skim milk solids" figure. If skim milk is fully replaced by whey, there will be a lowering in quality of the finished product. A partial replacement of skim milk by whey will tend to give sweet goods a softer texture and to retard the hardening or drying effects of aging. A replacement of the "skim milk solids" by whole milk products such as sweetened condensed whole milk will result in a product of marked superiority of flavor and texture.

**White Pan Breads.** All commercial white pan bread (round top) is produced by either the "straight dough" or the "sponge dough" procedure; the latter method is preferred by large wholesale bakeries because it is somewhat less sensitive to unavoidable variations, and produces whiter bread with more desirable eating qualities. The straight dough method is generally used in experimental baking since it is more sensitive to changes in ingredients or procedure, and it is frequently used by smaller retail bakeries and for specialty breads.

*Straight dough method.* In the straight dough method all the ingredients are measured and mixed together into a single dough. In the following formula for white pan bread the proportions of each ingredient are expressed in proportion to the flour, which is taken as 100.

White Pan Bread

| | |
|---|---|
| Flour | 100 |
| Water | 58–64 (Variable) |
| Yeast | 2–2½ |
| Shortening | 0–4 |
| Salt | 1½–2 |
| Sugar | 4–6 |
| Yeast Food | 0–½ |
| Skim milk solids | 0–6 |

For small-scale operation the following procedure may be used. The flour and shortening are scaled into the mixer while the salt, sugar, yeast, yeast food, and milk byproduct are added to the dough water (also known as the "absorption") and after thoroughly mixing, the solution is added to the flour and the mixing started. Generally three to six minutes mixing time will be necessary for small amounts of dough, depending on the type of flour, but for commercial size batches mixing time will extend to ten minutes or more. A smooth, extensible dough that is soft but that tears away from the hands without sticking is ideal. After mixing, the dough is scaled into pieces whose weight depends on the volume of the bread pans that are to be used; usually one ounce of dough per six cubic inches of pan volume is considered good practice. The properly weighed dough pieces are then given several "folds" and placed in suitable containers for fermentation. The total fermentation period for the straight dough procedure is divided into two periods of time; the first varies from 90 to 150 minutes and is carried out under normal atmospheric humidity at temperatures of 80 to 85°F. The second period is known as the "proofing period" and is generally about 60 minutes under conditions approximating 100°F and 95–100% humidity. Specially constructed "fermentation" and "proofing" rooms or cabinets are necessary and are available commercially. During the first period of fermentation, the dough is "turned" or "punched" at the end of 60 minutes and again at 30 minute intervals in order to expel carbon dioxide and stimulate yeast growth. Commercial equipment and schedules are usually adapted to local circumstances and time periods are varied depending on daily conditions. Between the fermentation and proofing periods, bread dough is allowed a so-called "bench time" which in large commercial production is the time required for dough to recover from processes of rounding and dividing of the dough by machine and usually approximates 20 to 30 minutes. In experimental work it is advisable to allow for these operations, and after the last punch, before panning, the dough is allowed to stand covered on the work bench for a period of 15 to 30 minutes. When the dough has "recovered" it is variously molded and placed in the baking pans. Then follows the proofing time and the bread is ready for baking. Depending on the type of oven and scaling weight of the dough, white pan bread is baked at 400 to 475°F, for 25 to 35 minutes. Bread is removed from the pans immediately after baking and is thoroughly cooled (30 minutes) before slicing and wrapping.

*Sponge-dough method.* For large commercial bread production, the "sponge-dough" procedure is chiefly used because of the improved grain, texture, and eating quality of the finished bread, as well as for the extended tolerances for variations in mixing, fermentation, proofing, and ingredients. Though rarely used in baking laboratories, the method is also adaptable to experimental practice and has the advantage of duplicating commercial conditions more accurately than the straight-dough process. In a typical sponge-dough process, the dough mixing is done in two steps. For mixing the "sponge," the following ingredients only are scaled into the mixer:

White Pan Bread (Sponge process)

| | |
|---|---|
| Flour | 60–70% (of the total amount) |
| Yeast | 1½–2 |
| Yeast food | ¼–½ |
| Water | 58–64 (variable) |

After mixing 4 to 10 minutes, depending on the size of the batch and the flour type, the temperature of the dough out of the mixer should be near 78°F. This is slightly lower than the dough temperature of straight doughs from the mixer. The resulting dough is called the "sponge." It is then placed in large fermentation containers (troughs) and allowed to ferment 2 to 4 hours (at approximately 85°F, and normal atmospheric humidity) omitting all punching and turning. During this time the sponge will increase to a maximum volume and then recede or "fall" indicating the sponge is ready for mixing with the remaining ingredients.

For mixing the final dough, the sponge is first transferred back into the mixer and the following ingredients added:

| | |
|---|---|
| Flour | 30–40 (Depending on % flour in sponge) |
| Sugar | 4–6 |
| Salt | 1½–2 |
| Shortening | 0–4 |
| Skim milk solids | 0–6 |
| Water | 58–64 (variable) |

The usual procedure is to reconstitute the non-fat dry milk with the dough water before adding to the other ingredients in order to facilitate complete hydration of the milk solids. Reconstitution is unnecessary with plain or sweetened condensed milk since the solids are already hydrated and dispersed. Another mixing period of the same length of time as the sponge mixing is generally necessary to produce the final dough of optimum handling properties. The temperature of the finished dough out of the mixer should be approximately 80°F. The dough is then permitted a fermentation period of 15 to 30 minutes (85°F, and normal humidity), after which follows the bench time, panning, proofing and baking in a manner similar to that described for straight doughs.

**Variety Breads.** A wide variety of special breads containing skim milk solids may be produced by following the general procedure previously described under the straight or sponge dough methods. Certain types of French and Italian breads do not require skim milk solids, but depend on very long fermentations for development of the desired flavors and characteristics, and no attempt will be made to describe their rather complicated production. The formulas given in Table 24 are expressed in percentage of the flour weight taken as 100, and their production variations are briefly described in the paragraphs following the table.

*Pullman and bun bread.* The doughs for these variety breads are made up in the same manner as regular white pan bread dough except that pullman bread dough is panned into standard pullman bread pans, and bun bread dough is divided by hand or machine into pieces of the desired size and placed on bun pans for proofing and baking. Pullman bread dough proofing is carried out with the covers in place, and the dough is proofed to such a height that the oven spring will bring the loaf up to the cover when the bread is baked. Usually a few trials are necessary before this rather precise stage is recognized.

*Whole wheat bread.* The percentage of whole wheat flour varies between 30 and 100 percent depending on the type of product desired. Flours of various strengths and granulations may be purchased. Usually the mixing time for whole wheat doughs is longer and the speed is slower than for white bread doughs since the gluten and starch require more time for moistening the coarser the whole wheat particles. For special flavors in whole wheat bread, various sweetening agents may be used including brown sugar, non-diastatic malt, syrups or honey. Whole wheat doughs do not have the same expansion as white bread doughs which thus permits a smaller amount of space for fermentation, a larger scaling weight, and a somewhat reduced oven temperature (420 to 440°F).

*Rye bread.* The percentage of rye flour may vary between 20 and 30 percent of the total flour, with variations in the types of bread being regulated by the type and proportion of various grades of rye flour. Special varieties of rye bread may be produced by addition of many supplementary ingredients: molasses, buttermilk, cultures or sauers, raisins, cheese, potato flour, onions, whisky mash, various combinations of spices, etc. The mixing of rye doughs should be longer and at a slower speed than white flour

TABLE 24. *Variety Bread Formulas*

| Ingredients | Pullman Bread | Bun bread | Whole wheat bread | Rye bread | Raisin bread | Sweet rolls |
|---|---|---|---|---|---|---|
| Flour | (patent) 100 | (patent) 100 | Whole wheat 100 | 1st clear 66<br>White rye 24<br>Dark rye 10 | (patent) 50<br>1st clear 50 | (patent) 85<br>Cake 15 |
| Water (Approx.) | 62 | 64 | 68 | 60 | 64 | 55 |
| Yeast | 2.5 | 2.5 | 2.5 | 2 | 2.5 | 3 |
| Malt | 1.5 | 1.0 | 1.0 | 2.5 | 1.0 | 2.0 |
| Salt | 2 | 2 | 2 | 2.25 | 2.0 | 2.0 |
| Sugar | 4 | 5 | (Molasses) 6 | 0 | 5 | 12 |
| Shortening | 4 | 5 | 3 | 1 | 3 | 12 |
| Skim milk solids | 6 | 6 | 4 | 4 | 4 | 6 |
| Yeast food | 0.25 | 0.25 | 0.25 | 0.25 | 0.25 | 0.25 |
| Caraway seed | 0 | 0 | 0 | 1.25 | 0 | 0 |
| Raisins, seeded | 0 | 0 | 0 | 0 | 35 | 0 |
| Eggs | 0 | 0 | 0 | 0 | 0 | 8 |
| Flavor | 0 | 0 | 0 | 0 | 0 | 0.02 |
| Temperature from mixer | 80°F | 80°F | 80°F | 75°F | 80°F | 80°F |
| Fermentation periods | 80/20 min. | 80/20 min. | 80/20 min. | 150/30 min. | 90/30 min. | 90/30 min. |

doughs, thus preventing the formation of sticky doughs that produce inferior products. The oven temperature should be approximately 390 to 440°F. Generally a small amount of "wet steam" in the oven during baking produces a desirable crust. Washing dough with water or starch solutions is sometimes done prior to baking. During proofing, the humidity should not exceed 80% and over proofing results in flat, unsightly loaves.

*Raisin bread.* Good bakery practice prescribes that a minimum of 50 parts of seeded raisins per 100 parts of flour meets consumer preference although a legal minimum of 35 parts raisins per 100 parts of flour is permitted. The raisins should be soaked in water at least two hours before adding to the dough during the last two minutes of mixing. Fermentation may be somewhat slower than with ordinary dough due to inhibitory action of the raisins and fermentation time should be slightly extended.

*Sweet rolls.* Standard sweet dough formulas differ from so-called "Danish pastry" in that the quantity of shortening is less and is incorporated during initial mixing rather than during special "roll-in" procedures characteristic of the latter type product. Variations are produced in a basic sweet dough formula by changing the topping, filling, flavor of icing, etc. Order of mixing ingredients greatly affects quality of the finished product and is best carried out in three steps: First, sugar, shortening, salt and flavor are blended; second, eggs are added slowly and mixed in thoroughly; third, yeast dissolved in water and flour are added together. Fluid or reconstituted milk may be used in place of water. Sweet dough should not be allowed to crust and if make-up is prolonged the dough should be kept covered.

**Cakes and Pastry.** In preparation of cakes, local differences are even greater than in the case of bread production. Consumer preference, economic considerations, local conditions and equipment, and formula variations all enter into the picture and no one formula or procedure can be described to cover all objectives.

Three separate types of cake usually recognized by the industry include the foam-type cake (angel food and sponge cake varieties), the white or yellow layer type cake (also loaf cakes and cup cakes), and pound cakes (wine cakes and other varieties produced without chemical leavening agents). Many variatons of each type are possible by the use of different proportions of ingredients, flavoring materials, fruits, nuts, etc. The use of shortening containing "emulsifying agents" rather than older types of commercial shortenings, permits a higher proportion of water and sugar, with respect to the same amount of flour, to be incorporated in the cake formula, thus producing the so-called "high-ratio" type cake. Before the improved shortenings were available it was difficult to produce a cake containing more sugar than flour, but cake formulas containing as much as

140 percent sugar, based on flour weight, are now possible, and this type seems to be preferred by the consumer because of the increased tenderness and improved eating qualities of the finished cake.

*Yellow layer cake.* The following yellow layer cake is the high-ratio type and is rich; the mixing procedure produces excellent texture and eating qualities in the finished cake, although a somewhat larger volume may be possible with other mixing techniques. A single-stage mixing, in which all ingredients are introduced into the mixer at one time, has been used to advantage, but the finished cake rarely has the improved texture of other mixing procedures. The quality of ingredients greatly affects the cake, and for best results only the highest grade materials should be used. All ingredients should be at room temperature while the temperature of the liquid ingredient should be such that the final cake batter has a temperaure of 70 to 75°F.

Yellow Layer Cake

| | | |
|---|---|---|
| Cake flour | 100 | Cream together at low speed, scraping down sides of mixer. |
| Shortening (emulsifier type) | 60 | |
| Sugar (finely granulated) | 115 | Sift together and add to mixer with enough liquid milk to form a stiff paste; mix 3 to 5 min. at low speed, scraping down sides of mixer. |
| Salt | 2 | |
| Baking powder | 6 | |
| Skim milk solids | 10 | Fluid or dried forms of milk may be used. |
| Water | 65 | |
| Whole eggs | 60 | Add remainder of milk and eggs slowly; mix 3 to 5 min. low speed. |

Egg whites may be used in place of whole eggs for white layers.
Batter is scaled into lightly greased pans and baked 25 to 28 minutes at 365°F.

The final batter should have smooth consistency, flow freely, and be at a temperature of 70 to 75°F. Nine to eleven ounces is generally satisfactory for seven-inch diameter pans. Oven temperature should be between 360 to 385°F with care being taken to insure a uniform, constant heat. "Flash" heat must be avoided to prevent cakes from too rapid crust formation while the interior of the cake remains unbaked. Finely granulated sugar is preferred in cake work since it dissolves rapidly, giving a smooth batter. Coarse sugar dissolves more slowly, resulting in a granular batter, and cakes of coarse grained texture. The undesirable curdling of cake batter, which may occur, is usually the result of improper temperature of ingredients. The slow addition of liquid ingredients and eggs also helps to prevent breaking of the cake batter emulsion.

*Cheese cake* offers one of the best opportunities for providing the consumer with variety in bakery products. With increasing popularity it promises to become a substantial outlet for soft curd cheeses such as cottage, pot and baker's cheese. The following formula may be considered a single type of which there are many varieties.

| | | |
|---|---|---|
| Bakers' cheese | 600 | Mix smooth for about 3 minutes. |
| Bread flour | 100 | |
| Salt | 5 | |
| Vanilla | 5 | |
| Shortening | 100 | Add and mix smooth. |
| Sugar | 25 | |
| Whole eggs | 200 | Add in three parts and mix smooth. |
| Milk (fluid basis) | 325 | Add slowly. |
| Egg whites | 350 | While beating egg whites add sugar slowly; beat to soft peak and then fold into the above mixutre. |
| Sugar | 75 | |

Baking temperature: 300°F.
Baking time: 75 minutes.

There is a wide variety of types, formulas and procedures for cheese cake, all requiring considerable practice with some variation to meet shop conditions. The formula and procedure given above are less complicated than many others and produce the standard type cheese cake. Slow baking is necessary to prevent excessive recession of the baked cake. It will rise to high volume during baking, but the recession will not be too great if oven temperature is kept around 300°F. Careful mixing is required at each stage to produce smooth, uniform batter which will bake into a cake of proper texture and eating quality.

*Pie crust* formulas are fairly uniform with the exception of the shortening content. The following formula may be considered representative.

| | |
|---|---|
| Pastry flour | 100 |
| Shortening | 40–70 (average 55) |
| Salt | 3 |
| Water | 30–35 |
| Skim milk solids | 2–4 |
| Sugar | 2–4 |
| Baking powder | 0.12–0.25 |

Special pie crust flours which are intermediate in strength between bread flour and cake flour are commercially available. A mixture of bread and cake flour is preferred if pastry flour is not available. The amount of shortening determines flakiness and tenderness of crust (amount of water is also important in this respect), although difficulty in handling usually limits the quantity used. In producing a high quality pie dough, overmixing should be avoided. Flour and shortening are "cut" either by hand or with special mixing blades until the mixture is in about one-quarter inch cubes. Water containing salt, sugar, milk, etc., is added slowly and mixing is continued only until the mass holds together. To avoid excess pan shrinkage and to facilitate handling, pie dough should be 6 to 8 hours old before rolling and baking. Oven temperature should be between 425 and 450°F, and baking time between 25 and 27 minutes.

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# 8

# CANNED PRODUCTS

## INTRODUCTION

Utilization of skim milk and whey in the manufacture of sterilized foods has not been one of the more important means for their disposal. Buttermilk is highly unstable toward heat and is not considered a suitable ingredient in sterilized products. Surplus milk and cream are sometimes canned and sterilized or they may be used as constituents of sterilized foods. During recent years many investigators have devoted time to the development of a method for the preparation of sterilized whole milk and cream which might compete with the fluid, bottled products. If acceptable products are eventually prepared, canned milk and cream might emerge from a surplus or byproduct classification to that of dairy products of first importance. However, the present rate of development does not indicate that this will occur in the immediate future.

Those canned sterilized foods which contain milk or its constituents, with the exception of standard evaporated milk, will be considered in this chapter. The manufacture of evaporated milk has been carefully described by Hunziker.[97] Milk, cream and whey will improve the nutritive value and flavor of many foods but in those foods which must be heat processed, special manufacturing problems arise. These problems will be discussed and possibilities for the development of new products will be indicated. Such a discussion must necessarily involve the behavior of the fat, protein and salts of milk. These consituents are present in varying proportions in byproducts from milk.

The United States Food and Drug Administration, Federal Security Agency, Washington, D. C., has not promulgated definitions and standards of identity for a majority of the products considered in this chapter. New products and modified dairy products which will be involved in interstate shipment should have label approval by the Food and Drug Administration.

The byproducts of milk are generally subjected to at least two heat treatments during the course of manufacture into sterilized or canned foods. One of these treatments is that of preheating or forewarming which always precedes concentration under vacuum, homogenization, sterilization and other phases of manufacture. The second heat treatment is the sterilization process itself. Special methods and pieces of equipment have been developed for preheating and for sterilizing milk and its byproducts.

## STERILIZING AGENTS

Agents advocated for food sterilization have included hydrogen peroxide and other chemicals, pressure, grinding, supersonic sound waves, high frequency electrical current and heat. Of the various methods, sterilization by heat is at present the only accepted commercial procedure. Many of the processes devised for the sterilization of milk do not produce a sterile product but one in which bacterial growth is temporarily retarded.

**Chemical Sterilization.** Milk "sterilized" with oxygen by the Hofius process kept only 3 days at 68°F.[5]

Hydrogen peroxide has been used from time to time as a sterilizing agent. The Italian process developed during World War II consisted in treating milk with 2 percent by weight of 39 percent hydrogen peroxide and allowing the milk to stand 8 hours.[40] The number of milk bacteria attained a minimum 12 hours after addition of the peroxide and thereafter increased. Other processes including the use of either oxygen or carbon dioxide under pressure have been tried.

The use of certain antibiotics for the sterilization of milk has been investigated.[38] Streptomycin was uniformly ineffective in killing spores while penicillin was effective only against certain species.

**Pressure.** Hydrostatic pressure has been investigated as a bacteriostatic agent[187] and it has been observed that pressures up to 5000 pounds per square inch slowed the rate of denaturation of serum globulin.[103] Although many common bacteria become permanently unable to reproduce after exposure to high pressures (600 atmospheres), sterilization by pressure is at present only of scientific interest.

**Electronic Sterilization.** Electronic sterilization of milk and other foods at room temperature or even lower is being approached through the use of cathode rays.[22, 47, 49] The reports indicate that electronic bacterial destruction in milk can be attained. But there is some question whether undesirable side reactions that tend to impair the flavor, nutritional and physical properties of the product can be eliminated. Experimentally milk has been placed in a petri dish, in airtight plastic capsules or in other suitable containers. It is then subjected to bombardment by electrons discharged at a potential of several million volts, but for periods of time as short as a thousandth of a second. Electronic destruction of bacteria has been observed at temperatures below minus 300°F. Commercial application of this type of sterilization to milk products must await the results of further research.

**Heat.** Heat is firmly entrenched as the best agent for sterilizing food. Steam and hot water are universally used as heating media but heating by electricity has had commercial application in milk pasteurization.

## METHODS OF HEAT PROCESSING

**Milk Products Adaptable to Heat Sterilization.** Fluid food preparations containing milk or cream require varying degrees of heating for their manufacture or preservation. These foods may be divided into two classes, (a) foods cooked and served at once, and (b) foods processed in sealed containers for storage and future use. Foods of the first class are prepared in restaurants and homes and present problems similar to those of the second group though simpler. While this discussion will be concerned with the manufacture of canned, heat sterilized foods, the principles involved apply also to those foods which are cooked and served in bulk. More freedom may be exercised in the choice of ingredients and in the quantities used and fewer processing difficulties will be encountered when the food is not to be processed in cans.

The byproducts of milk which are adaptable to sterilization in hermetically sealed containers may be classed in three groups.

*Special milk products.* These are modified dairy products which may contain such constituents as chocolate, sugar or stabilizers. Their reaction is substantially the same as that of milk, pH 6.5 to 6.6. The group includes modified infant foods, sterilized milk drinks, canned cream, and evaporated skim milk.

*Milk-vegetable products* comprise the group which includes cream soups, sauces and vegetables. These are non-acid products with a reaction between that of milk and the vegetable used. The term "cream" as applied to cream soups or vegetables signifies a product made with milk or its products and thickened with flour, starch or some other suitable thickening and binding agent. The term "cream-style" should be used when skim milk or whey products are substituted for milk.

*Whey-fruit products* contain sugar and stabilizer in addition to fruit and whey solids. The reaction of the mixtures may approach the pH of the fruit. These are experimental products and include whey-fruit beverages and whips. It is hoped that their consideration here may stimulate research and commercial development.

**Preheating.** *Types of steam heaters.* The hotwell or heating vat, after years of use, is gradually being supplanted by continuous tubular heat exchangers. Hotwell heating is generally accomplished either by injecting steam directly into the milk product or by means of a steam-heated jacket built around the vessel. Steam injection is a rapid and efficient means of heat transfer but it is not the best method because of the possibility of contamination with water-borne impurities and because the steam condensate dilutes the milk. Several arrangements for steam injection heating including the Grindrod processes[68–81] have been developed.[35, 139, 183]

The usual type of internal tubular heat exchanger has been supplemented in recent years by new heaters, some of which are capable of raising the temperature of fluid milk products to 320°F. These heaters force relatively thin layers of milk over heated surfaces at very high velocities. The rapid flow of the milk product helps to retard the formation of milkstone on the heating surface. Two of the early heaters of this type were the Nielson[110] and Mollinger[123] heaters. The original high velocity Mallory heater[34, 114] consisted of a high pressure pump and many feet of 0.18 inch I.D. stainless steel tubing mounted in a steam chest. Liquids were heated in a few seconds to 320°F and cooled rapidly by passage through similar tubing. The Mallory heater is still constructed with small diameter tubes but these are connected by headers to facilitate cleaning.[184]

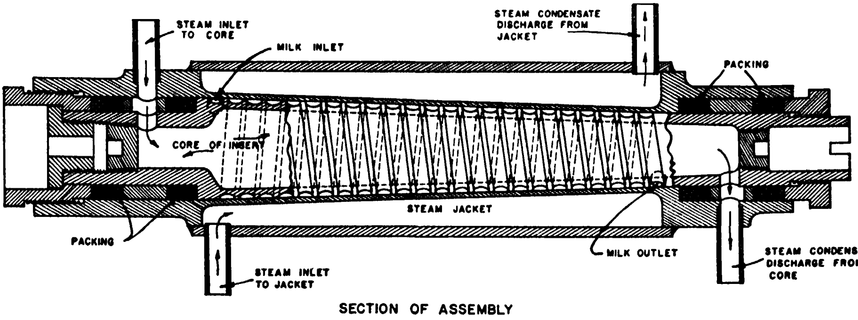

Fig. 13. Assembly drawing of Bureau of Dairy Industry heat exchanger, showing helical-coil heating area and inlets and outlets for milk and steam.

Another heater with high velocity flow characteristics has been designed by Hanrahan.[82] A drawing of this helical coil heater is shown in Figure 13. The heater operates at much lower pressures than the Mallory unit and it can be readily disassembled for cleaning.

North has patented a rapid[131] flow heat exchanger which consists of several straight half-inch tubes connected with headers which can be opened for cleaning. The liquid is rapidly circulated and recirculated until the desired temperature is reached. The flow at intake and outlet are equalized to maintain continuous operating conditions.

Rapid-flow high-temperature heat exchangers are still in the experimental stage but commercial size equipment is gradually being developed. At least two different units are now available.

*Milkstone* formation during the preheating of milk is the gradual deposition of milk solids on the surfaces of the heating unit. It is not to be confused with the complete gelling of the protein system which takes place during coagulation. Milkstone, being composed largely of organic matter with some calcium and phosphorus, seriously interferes with heat transfer. The rate at which it deposits in the tubes of heat exchangers largely determines the length of time the heater can be operated before cleaning becomes necessary. High velocity flow through small heating passages will to a limited extent retard milkstone formation. However, even under the most favorable flow conditions, such as are used in the Mallory heater, an objectionable coating will form in a few hours if fresh, cold milk is heated to 212°F or higher. Bell and Sanders[17] have shown that milkstone formation occurs largely at those temperatures that are favorable to the coagulation of albumin. The burned deposit on the tubes of a high-temperature short-time heater was greatly decreased by preheating the milk or whey to 190 or 200°F. before passing it through the heater. Data plotted in Figure 14 shows the relation between the temperature and time of heating which is necessary to coagulate albumin. Since milkstone formation is associated with the coagulation of albumin, equipment should be designed and operated so that the milk is exposed to the heating surfaces for as short a time as possible. This can be done by the use of high velocity flow through the heater and by providing a suitable holding pipe, vat or hotwell at the point of maximum albumin coagulation. A holding unit for milk at temperatures of 170 to 200°F inserted between tubular heaters operating below and above a temperature in this range will collect much of the deposit which otherwise would form in the heater tubes. Hotwell heating by jacket or direct steam in the range 145 to 210°F will cause the milkstone to form where it will be least objectionable. Crighton has developed a combination process[35] which employs a tubular heater to quickly heat milk to 190°F, then a direct steam injector to raise the temperature as high as 280°F Finally a pressure hotwell acts as a holding tank and a depository for much of the precipitated milkstone.

*Electrical heating* of milk and other food products has attracted much attention in recent years. Some success has been attained in the use of normal electric heating to bring milk from regenerator to pasteurization temperature. There is perhaps more promise in the still experimental use of radio-frequency heating for pasteurization, preheating or sterilization of milk products and other foods. When a high-frequency current is passed through milk, heat is generated very rapidly within the liquid and there are no heating surfaces to become coated with milkstone. This almost instantaneous rate of heating is the most distinctive characteristic of radio-frequency heating. In steam heating of milk the time required to raise the temperature to the desired degree may be long enough to cause undesirable changes in the product.

Brown, Hoyler and Bierwirth[24] heated milk in a glass tube in a freely-falling stream from 140 to 205°F in 0.067 second. The milk was cooled by vacuum expansion to 135°F in 0.2 second. No trace of cooked flavor was noted. The metal electrodes were outside the glass tube and not in contact with the milk. Although dielectric heating is very rapid, the problem of rapid cooling remains. In the above experiments the cooling problem was solved by vacuum expansion which might not be possible under all kinds of operating conditions. High frequency heating requires expensive equipment, the cost of the energy needed is high and the methods are still in the laboratory stage of development.

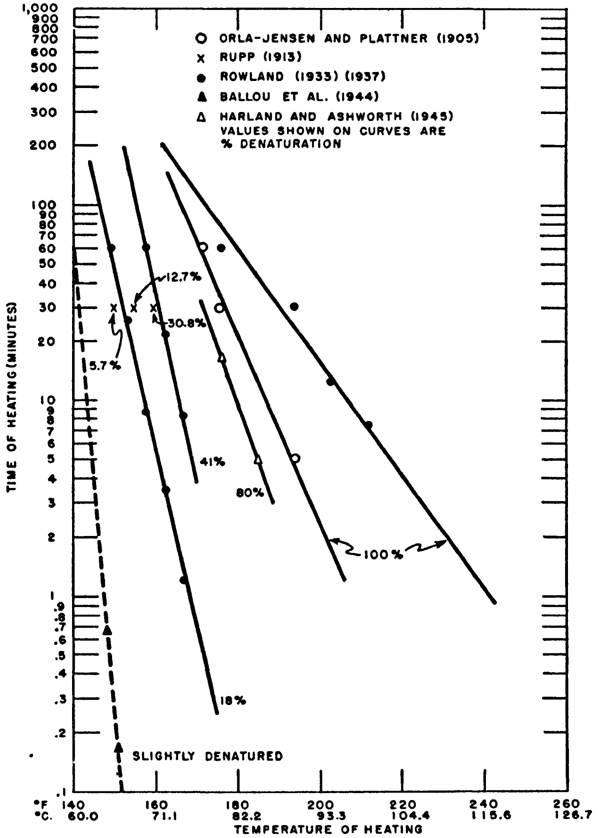

Fig. 14. The effect of time and temperature of heating the albumin-globulin fraction in milk upon the rate and extent of its denaturation. (The broken line marked "slightly denatured" indicates the time required for the first appearance of turbidity in a 25% human serum albumin solution, including 2% globulin, according to Ballou et al.[16] The solid lines are drawn from data of Rowland,[6, 9] Harland and Ashworth,[85] and Orla-Jensen and Plattner[132]; the three indicated points are from Rupp.[145])

**Sterilization.** Many methods for the preservation of foods have been developed and patented. Sterilization by heat, generally under pressure, is still the most satisfactory process for the preservation of liquid foods, although sugar is an important preservative in the milk industry. The advances which have been made in sterilization processes have been reviewed by Ball[11] and by Jackson and Benjamin.[100] The methods used for dairy products can be conveniently divided into two parts: (a) autoclave sterilization in which the product is sealed in the container before it is heated, and (b) sterilization in bulk, followed by aseptic packaging. High-temperature short-time processes have been devised for both autoclave and bulk sterilization methods.

*The autoclave* is the basic piece of equipment for the heat processing of foods. Its mechanical and operational features have been constantly improved so that each container receives the same heat treatment. Means have been provided to rapidly heat to and cool from the holding temperature. The revolving reel autoclave now in use for processing evaporated milk and other sterilized milk products was developed and patented by Meyenberg in 1884.[118] The uniform agitation of the cans provided by Meyenberg's invention shortened the time required to reach the sterilization temperature and equalized the heat treatment received by the product in different cans.

The Anderson-Barngrover[98] continuous sterilizer is a modification of the usual autoclave equipment. The cans of product are conveyed through two preheating units, into a pressure sterilizing compartment and then to a cooling unit. The equipment operates automatically and eliminates the heavy time-consuming work of loading and unloading the batch type autoclave.

Agitating devices of various kinds[15, 21, 83, 107, 108, 133, 182] have been developed in recent years so that high-temperature short-time sterilization may be conducted in a modified autoclave. The canned product is subjected to a violent agitation which causes it to come up to the sterilizing temperature, generally between 255 and 280°F, in about 2 minutes. A holding period of 1 to 3 minutes and rapid cooling complete the process in a total time of 6 or 7 minutes. The effect of agitation during sterilization on the viscosity and body of milk products will be discussed in the section on physico-chemical problems.

*Sterilization followed by aseptic packaging* of milk products has been attempted by several methods. Sterilization in bulk can be accomplished more rapidly and with fewer detrimental changes in the food than is the case with processing the container. But the problems of commercial aseptic packaging have been difficult to solve. In many of the patented processes it is assumed that bacterial contamination will occur during packaging and provision is made for mild sterilization of the packaged product.

Steam injection sterilization of bulk milk products was tried by Grindrod.[68–81] It has also been practiced by forcing steam directly into the can containing the food and subsequently sealing the can. Ayres and Lang[8] applied this process to milk products, but most of the patents covering similar methods are concerned with other foods.

A complete process for sterilizing empty containers and covers, sterilizing the food in bulk, and putting it into the sterile containers and sealing the containers under aseptic conditions has been devised by Ball.[14] A pilot plant, flash sterilization and aseptic filling process is described by Martin.[115] The Grindrod patents previously cited described equipment and processes for the sterilization of dairy products by subjecting them to the action of high-velocity steam jets. After sterilization the material is cooled and the moisture added as condensed steam is removed by evaporation under vacuum. The processes of McClatchie[116] and of Hass[86] for evaporated milk are somewhat similar to that of Grindrod.

Fluid milk products may be readily sterilized in bulk by the use of the high-temperature short-time heat exchangers discussed in the section on preheating.[82, 110, 114, 123, 131] A device for aseptically collecting experimental samples from the Mallory heater has been described by Curran, Bell and Evans.[37]

Electrical heating under pressure in a specially designed autoclave but without provision for aseptic packaging is the subject of one patent.[146]

*Time and temperature relationships* during sterilization determine the sterility and, in fluid milks, the protein stability and flavor changes which occur during storage. The conventional autoclave method of sterilizing fluid milk in the container for 15 to 20 minutes at 240 to 242°F yields a commercially sterile product which undergoes few storage changes. For cream-style foods the sterilization time must be extended. The process time for most soups in quart containers should be 50 minutes at 250°F (121.1°C). A condensed soup in No. 1 cans should be held at least 30 minutes at 250°F.[26] Since the size and type of container in which the food is sterilized is important, a study has been conducted of the comparative rates of heat penetration in cans and in glass jars.[161]

The production of a line of creamed vegetables should be preceded by experimental work since there is no extensive commercial experience which may act as a guide to proper processing.

Detailed bacteriological studies have indicated the time-temperature relationships which should be used for different foods when they are sterilized in various size cans. Processes for non-acid canned foods which contain no milk constituents have been compiled and tested by the National Canners Association.[127]

Ball has shown how the problems of thermal processing in canned foods[13] and in milk pasteurization[12] may be solved mathematically.

Stumbo in an analysis of process evaluation presented a method for ascertaining the location in the container where the probability of bacterial survival was greatest.[153] He demonstrated that lethality of heat treatment at the point of greatest temperature lag in the container was not always an indication of the sterilizing capacity of the process.[154]

High-temperature short-time sterilization is preferable to normal autoclave processing from a bacteriological as well as a flavor viewpoint. After six years of extensive experiments Ball[11] found:

> For four processes at higher temperatures having equal effect on quality of evaporated milk, the calculated ratios of sterilizing value, referred to the 115.6°C (240°F) process, are:

| | |
|---|---|
| 126.7°C (260°F) | 2.2 |
| 132.2°C (270°F) | 2.8 |
| 137.8°C (280°F) | 3.7 |
| 143.3°C (290°F) | 4.4 |

It is of interest to note that the time-temperature relationships for the destruction of bacterial spores in milk are of the same nature as the relationships between the time and temperature of coagulation of milk. Both are logarithmic with respect to time. The data of Curran, Bell and Evans[37] showing the rate of destruction of spores in milk heated in a high-temperature short-time heater, is plotted in Figure 14 (dotted line). The test organism was a sporing species isolated from spoiled, commercially canned, evaporated milk. The thermal-death-time curve is a straight line with a slope of 23°F per logarithm cycle.

Townsend, Esty and Baselt[160] in studying the spores of *Cl. botulinum* found that the slope of the thermal-death-time curve varied in different food media but was about 16 to 18°F per logarithm cycle. The heat coagulation curves show a rate of 30°F or more per cycle. The position of the thermal-death curves, relative to the heat-stability curves, indicates that spore destruction can be accomplished with less danger of coagulating the milk at high rather than at low heating temperatures. Also, it should be possible to sterilize most milks containing up to 23½ percent solids-not-fat without coagulating them. At higher concentrations coagulation might occur before sterility was attained.

The time and temperature of sterilizaiion of fluid milks have an important bearing upon the stability in body and flavor of the product during storage. Milks sterilized by the usual autoclave methods are relatively stable in storage. But when it became possible to process milk by quick heating methods it was soon found that undesirable changes occurred early in the storage period. High-temperature short-time sterilized milks immediately after processing resemble pasteurized milk, but during storage at room temperature a thickening in body and a stale or oxidized flavor develop.[11, 17] This physicochemical instability appears to consist of a gradual, irreversible denaturation of the casein. After flash sterilized skim milk is held 6 to 8 weeks at room temperature there is a precipitation of the protein, or thickening, and the formation of a gel structure at concentrations of 18 percent or more milk-solids-not-fat.[176] The degree of precipitation, thickening or gelation seems to be proportional to the concentration of non-fat milk solids. Flash sterilized cream apparently does not show a protein change during the first year of storage.

The development of a stale flavor has been observed in flash sterilized milks. The intensity of this flavor, which is similar to the stale flavor of dry milk, increases with storage time. Since the flavor does not seem to appear in quickly sterilized cream it may be associated with the protein fraction of the milk. Oxidized flavors sometimes become objectionable in the high-temperature short-time products.

It is of interest to note that these body and flavor changes do not occur in milks which are heated by the older and more severe methods. When means are discovered to give the flash sterilized products the physicochemical stability of the conventionally heated products, a great improvement in heat processed dairy products will have been made.

## PHYSICOCHEMICAL PROBLEMS

The important physicochemical changes which are associated with the heat processing of milk products are heat coagulation and the development of body, flavor and color. Several agents may be used to coagulate milk but rennet, acid and heat are those most frequently encountered. Rennet coagulation, which occurs as a gradual formation of a gel structure, is basic to cheesemaking. Acid coagulation, a sudden precipitation of milk casein, is used in casein manufacture. The heat coagulum of milk usually forms slowly during heat treatment and, if it is allowed to proceed, a gel structure similar to that caused by rennet is formed.

**Heat Coagulation.** It is well recognized that the heat coagulation of protein consists of two distinct processes, denaturation and flocculation. Denaturation consists of a little understood change of the protein during heating. This precedes actual agglutination which is brought about by neutralization of the particle charge. Coagulation of the proteins in milk is dependent upon the rate of their denaturation since the milk salts furnish an ever-present supply of electrolytes to flocculate the denatured protein.

There is little experimental evidence to indicate whether the heating of the calcium caseinate-calcium phosphate complex in milk produces changes in the protein before the occurrence of those changes involved in the coagulation process. Svedberg, Carpenter and Carpenter[155] observed a doubling in the molecular weight of pure casein when it was heated to 104°F. However, Nichols et al.[130] found no difference in the particle-size distribution curves of the casein complex in skim milks separated at 104°F and heated to 149 and 203°F. Viscosity change may be used as a criterion for measuring changes in the caseinate-phosphate complex during heating.[50] There was no significant increase in viscosity as a result of the heat treatment used to concentrate skim milk, but sterilization without coagulation did cause a viscosity increase when the milk was diluted and compared with the original product. Wright[185] in studying racemization velocities concluded that the chemical nature of the casein complex in milk was not changed by heating at 248°F for 30 minutes. However, evaporated milk which has thickened but not coagulated during sterilization undergoes a marked thinning in the first few days of storage after its manufacture. This decrease in viscosity is probably the result of a decrease in the water binding capacity of the sterilized protein. Whether this has occurred as a result of molecular changes caused by the heat treatment is unknown.

Heat coagulation is influenced by: (a) the magnitude of the heat treatment; (b) the concentration of milk-solids-not-fat; (c) the acidity; (d) the ionic equilibrium; and (e) the presence of un-ionized and inert material. Heat stability, or the resistance of the milk protein to coagulation, is expressed as the time in minutes which is required to start coagulation at a designated temperature.

*Magnitude of the heat treatment.* When fresh milk is heated above 140°F for 5 hours there is some coagulation of lactalbumin according to Orla-Jensen and Plattner.[132] However Rupp[145] found no albumin was coagulated when milk was heated for 30 minutes at 145°F. The heat denaturation of the albumin-globulin fraction in milk was determined analytically at different temperatures by Rowland.[142, 143] Data of these investigators together with data of Ballou et al.[16] have been plotted in Figure 14.

The change in the slope of the curves as denaturation progresses indicates that to obtain a given bacterial destruction less albumin and globulin will be denatured when high-temperature short-time heating is used than when slower methods are employed. A comparison of Figures 14 and 15 shows that less heat is required to coagulate the albumin and globulin fraction of milk than is needed to destroy bacterial spores or to coagulate milk samples of less than 38 percent solids-not-fat.

Milk is not considered to be coagulated until the calcium caseinate-calcium phosphate complex is precipitated or a gel structure is formed. This change requires much more heating than is necessary to denature the albumin alone. The heat needed to coagulate fresh milk varies from an exposure at 240°F for 120 to 300 minutes, to heating at 320°F for a few seconds.

Milk is subjected to two distinct heating processes in the manufacture of canned products. These processes are forewarming and sterilization.

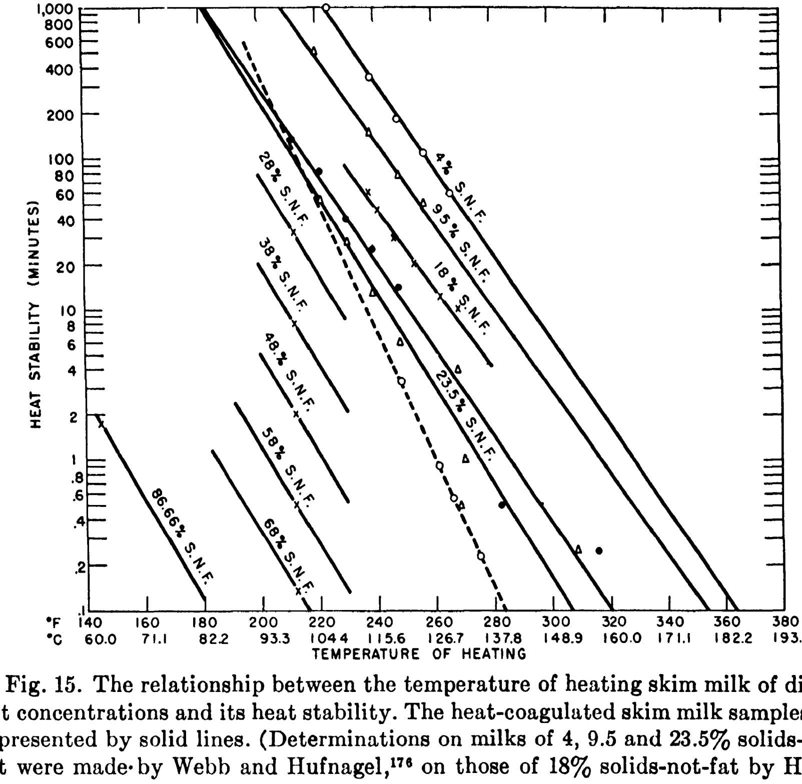

Fig. 15. The relationship between the temperature of heating skim milk of different concentrations and its heat stability. The heat-coagulated skim milk samples are represented by solid lines. (Determinations on milks of 4, 9.5 and 23.5% solids-not-fat were made by Webb and Hufnagel,[176] on those of 18% solids-not-fat by Holm, Deysher and Evans,[92] by heating the milk in cans in a pilot sterilizer, except for points at 23.5% below 4 minutes, which were obtained by passing the milk through a Mallory heater.[170] Wright[186] determined the values at 28 and 38%, calculated those at 48 to 68%, and extrapolated low temperature values to obtain the line at 86%. The dotted line is a thermal death time curve for test spores heated in milk by means of a Mallory heater and was determined by Curran, Bell and Evans.[36])

Forewarming is the process of applying heat to milk in order to increase its heat stability after concentration but before sterilization. When heat is applied to milk at the time of forwarming its heat stability is lowered unless it is concentrated to more than 13.5 percent milk-solids-not-fat.[173] The effect of forewarming upon the heat stability of skim milks after concentration from 9 to 18 percent solids is shown in Figure 16.[173] The critical concentration beyond which forewarming improves heat stability varies with different milks between approximately 10.5 and 14 percent solids-not-fat. This indicates that the forewarming of milk for condensing or drying should be done at low temperatures (below 160°F) if the product is to be used in a sterilized food at concentrations of less than 10.5 percent milk-solids-not-fat. The presence of other destabilizing factors sometimes overshadows the effects of forewarming temperatures. This is the case in the cream soups, where coagulation of the milk protein occurs regardless of the forewarming temperatures used in processing the milk.

Within the range 11 to 14 percent milk-solids-not-fat the forewarming temperature appears to be unimportant as a means of controlling heat stability. However, temperatures of forewarming to 212°F[41, 111, 152] and beyond[170] the boiling point increase the heat stability of products subsequently concentrated to 14 percent or more milk-solids-not-fat. Evaporated skim milk which contains 18 to 20 percent m.s.n.f. is generally adequately heat stabilized when the fresh milk is forewarmed at temperatures between 190°F and boiling.

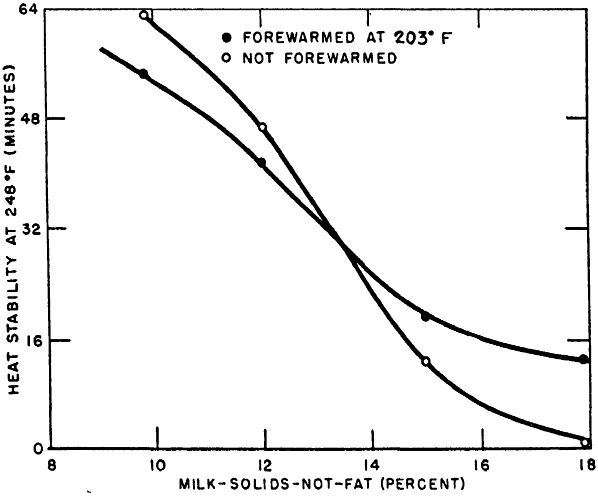

Fig. 16. The effect of forewarming upon the heat stability of a skim milk at different concentrations.

If milk is forewarmed and concentrated in a subsidiary plant, then shipped to the main plant and mixed with an equal quantity of fresh milk, high temperature forewarming of the mixed milk will usually decrease the heat stability of the final concentrate.[140]

The relation of time to temperature of coagulation in skim milk of different concentrations has been found to be logarithmic with respect to time.[92, 173, 186] Quantitative studies of this relationship have been made and some of the available data are plotted in Figure 15. It is not to be expected that two different milks of the same concentration will always yield points on identical curves. The two curves representing milk of 23.5 percent solids were obtained from samples having different heat stabilities.

Cole and Tarassuk[32] have studied the relationship between temperature and time of coagulation in cow and goat milk. They found that while most samples of milk showed a fairly straight-line relationship between coagulation temperature and the logarithm of coagulation time, there was considerable variation from this relationship over a temperature range of 230 to 320°F. It seems probable that the relationship is logarithmic but that experimental difficulties, which cannot yet be controlled, account for lack of agreement between investigators.

The slopes of the heat stability curves of Figure 15 vary from 30 to 36°F for each logarithm cycle through which the lines pass. This is in contrast to the more rapid initial coagulation of albumin and globulin in milk shown in Figure 14.

The curves for the coagulation of 18 and 41 percent denatured albumin and globulin pass through 10°F per logarithm cycle. The rate changes however, for at 100 percent denaturation the slope varies between 21 and 36°F per cycle.

*Concentration of milk-solids-not-fat.* Experimental observations indicate the quantitative relationship between the concentration of non-fat milk-solids and heat stability to be linear. Holm, Deysher and Evans[92] found that in the concentration region, 18 to 20 percent milk-solids-not-fat, the change of coagulation temperature for every 1 percent change in concentration was approximately 2.7°F. If from the data of Figure 15, the coagulation temperatures at 1 minute of heating are plotted against the concentrations, the best straight line which can be drawn through the points on the graph will show that for every 1 percent change in concentration there is a temperature change of 2.2°F. This figure was drawn from the data of several investigators and covers the concentration range, 4 percent to 86 percent milk-solids-not-fat. It is probable that natural variations in the heat stability of different milks and in the use of different experimental methods would cause this value to vary between about 1.8° and 2.7°F for each 1 percent change in solids content of the skim milk.

The heat stability-concentration relationship in milks may differ from that which could be determined in pure colloidal suspensions. Whenever the concentration of milk protein is changed by dilution of the milk, the dilution also alters the number of active ions, the conditions governing adsorption, and the stability of the suspended phase. Ballou et al.[16] working with aqueous dilutions of human serum albumin found that thermal stability seemed to vary inversely with the protein concentration. Chick and Martin[30] found in the case of solutions of hemoglobin that the rate of coagulation was proportional to the concentration of residual hemoglobin.

*The effect of acidity* on the heat coagulation of milk is generally a destabilizing one. While the degree of natural acidity which occurs in fresh milk does not appear to be related to heat stability, acid from bacterial growth, from prolonged heat treatment or from direct acid additions has a marked effect upon coagulation during heating.

Small changes in acidity within the approximate range pH 6.52 to 6.65[20, 165] produce effects similar to the changes in salt balance which will be discussed later. Large acidity changes which bring the pH below this range generally destabilize the milk. Such a change, caused by bacterial growth, was shown by Rogers, Deysher and Evans[141] to lower heat stability. The addition of acids directly or of acid foods such as tomato juice will quickly destabilize milk if its reaction drops below pH 6.54 to 6.50. No critical pH value can be given but a small shift from the original pH of the milk will often sharply lower heat stability.

The hydrogen-ion content of a milk is increased by concentration of the milk. The magnitude of this increase for skim milks has been determined by Howat and Wright[96] who found a milk of 10 percent solids to have a reaction of pH 6.6 but at 30 percent it was pH 6.20 and at 65 percent, pH 5.97. It is pointed out that the lowering of the pH which accompanied milk concentration inevitably produces conditions favorable to heat coagulation.

The investigations of Whittier and Benton[178, 179, 180] and of Gould[62, 63] on the effect of heating milk upon acid production are of particular interest among many papers on this subject. The rate of acid formation in milk is approximately proportional to the time and temperature of heating. The acid is formed from the lactose in the milk and is quantitatively related to the amount of lactose in the sample. Gould found 25 to 30 percent of the lactose in milk was destroyed during heating to 241°F for 2½ hours. Under these conditions the reaction changed from pH 6.4 to 5.8. Formic acid constituted about 80 percent of the total volatile acids whereas only 5 percent of the total acid produced was lactic acid. Since acid formation is a direct function of time and temperature of heating and of lactose concentration, care should be used in the pre-sterilization heat treatment of milks to avoid the production of enough acid to destabilize the product.

*Ionic equilibrium.* When milk is heated changes occur in the salt equilibrium of the system which probably affect heat coagulation. Söldner[151] and subsequently many other investigators have observed that calcium phosphate is precipitated in the heating of milk. Leighton and Deysher[111] have shown that there is a precipitation of calcium and magnesium as citrates and phosphates with the absorption of heat during coagulation.

Howat and Wright[94, 95] studied the heat coagulation of calcium caseinate solutions heated at 248°F for periods up to 5 hours and concluded there was a dephosphorization of the molecule preliminary to coagulation. Ramsdell and Whittier[137] present evidence that the calcium of the calcium caseinate-calcium phosphate complex is combined through the phosphate groups. If both calcium and phosphorus are set free during heating, there would be slight, if any, increase in the calcium-ion concentration of the solution. The effect of ion-exchange treatment upon the heat stability of skim milk is discussed in Chapter 9.

Sommer and Hart[152] first clearly showed that the heat stability of milk could be greatly affected by changing its "salt balance." Their work, together with the work of Mojonnier and Troy,[122] was of great assistance in the control of heat coagulation in evaporated milk manufacture.

The effect of changes in the salt equilibrium upon the heat stability of milks of various concentrations has been studied by other investigators.[20, 48, 120, 173, 177] The quantities of positive or negative ions required to produce large changes in the heat stability of a milk may sometimes be so small that they cannot be determined analytically. Significant changes in hydrogen-ion concentration, however, will generally be reflected in pH values. An instability of milk at normal pH considered to be due to an increased calcium-ion activity was corrected by administration of sodium citrate to the cows producing such milk or by adding the citrate directly to the milk.[148]

Plain or dilute milks may react differently toward ionic changes than do their concentrated products. Most skim or whole milks of normal concentration are decreased in heat stability by the addition of salts containing strong positive ions such as $H^+$, $Ca^{++}$, or $Mg^{++}$. The salts of the effective negative ions, $OH^-$, citrate, phosphate, and bicarbonate, stabilize these milks. Sometimes an unconcentrated milk is stabilized by strong positive ions and destabilized by those of opposite charge, but this is uncommon. After concentration to a solids content greater than 14 percent, the milks can generally be stabilized by the same salts which were used for stabilization at normal concentration. But sometimes milks which were stabilized by strong netative ions before concentration require the addition of positive ones for stabilization after concentration. Every milk represents a different colloidal system of which the heat stability and salt balance characteristics must be experimentally determined. This can be readily done by adding different quantities of suitable salt solution to test cans of the milk. The cans are then sealed, sterilized, and examined for heat stability and body characteristics. Calcium chloride and calcium acetate are commonly used as a source of positive ions and disodium phosphate or sodium citrate furnish negative ions. Occasionally a milk is found which is naturally balanced for maximum heat stability and is destablized by the addition of any salt.

*The effect of inert and un-ionized material* on heat coagulation will be considered to include the effect of fat, sugars and starches, fruit and vegetable pulp and similar substances. All of the special foods discussed in this chapter contain some of these ingredients. Insert materials appear to promote protein coagulation by acting as nuclei which attract, adsorb or mechanically hold together the protein molecules. This localized concentration of protein seems to have the same destabilizing effect as an overall concentration increase. Large aggregates or groups of aggregates could provide a destabilizing physical structure about which coagulation might proceed. The destabilizing influence of carbohydrates may be due to the mechanical effects of large molecules.

It is of interest to note that Howat and Wright[95] found visible coagulation usually did not occur until 50 to 60 percent of the protein in a calcium caseinate solution was rendered insoluble during heating. It can be assumed from this that if the protein, as it was denatured, was able to gather on inert pulpy material or in large molecules of un-ionized carbohydrate, mass coagulation could be accelerated.

The presence of fat as it is normally dispersed in milk lowers the heat stability of the product but slightly.[41, 92] However, homogenization increases the dispersion of the fat, creates new surfaces for protein adsorption, causes clumping of the globules, and decreases heat stability. The stability is lowered somewhat in proportion to the severity of the homogenization treatment. During the process the fat globules are reduced to less than one-half of their original diameter. This causes a five-to-tenfold increase in new fat surface and a hundred-to-thousandfold increase in the number of globules. Most of the fat globules in a homogenized product are reduced to less than two microns in diameter.

The homogenization process has been studied by many investigators. Some have been especially interested in the effect of homogenization on protein stability.[25, 28, 41, 44–46, 91, 162, 163, 166, 167, 171]

The feathering of cream in coffee is an indication of low heat stability. The manufacturing processes and heat treatments which produce high heat stability in a cream also produce cream of low feathering tendencies.

The viscosity of homogenized cream bears an inverse relationship to its heat stability. Creams which are homogenized under conditions which are conducive to the development of high viscosity are relatively unstable to heat and may show a tendency to feather in coffee.

The effect of the specially prepared inert materials, tomato pulp, ground filter paper, a fine decolorizing carbon, and coagulated, dispersed casein, upon the heat stability of plain and condensed skim milk is shown in Table 25.[176] The addition of these materials to skim milk did not change its reaction. They merely acted as nuclei to promote coagulation of the protein.

TABLE 25. *The Effect of the Addition of an Inert Phase on the Heat Stability of Skim Milk*

The 6% solids milk was prepared from fresh, raw skim milk by dilution with the suspension of the inert material. The 18% solids milk was prepared by forewarming skim milk to 203°F for 10 minutes, condensing it to 30% solids and diluting with the aqueous suspension.[176]

| Skim Milk | | Inert Phase Added to Milk | | Heat Stability of Milk at 240°F, min. | Decrease in Heat Stability, % |
|---|---|---|---|---|---|
| Sample No. | Concentration of Solids, % | Kind | Concentration, % | | |
| A1 | 6 | None—Control | — | 190 | 0 |
| A2 | 6 | Tomato pulp, centrifugally separated, washed and adjusted to same reaction as milk | 1.0 | 158 | 17 |
| A3 | 6 | Filter paper, ground in 95% alcohol for 35 hrs. in a ball mill, filtered, washed, boiled in water, added to milk | 1.0 | 150 | 21 |
| B1 | 18 | None—Control | — | 78 | 0 |
| B2 | 18 | Decolorizing Carbon | 0.5 | 32 | 59 |
| B3 | 18 | Decolorizing Carbon | 2.0 | 20 | 74 |
| B4 | 18 | Fine dispersion of Hammarsten Casein | 0.3 | 30 | 62 |
| B5 | 18 | Heat coagulated casein from roller dried skim milk. Casein, centrifugally removed from water suspension and washed. | 2.0 | 20 | 74 |
| B6 | 18 | Same casein as B5 but the casein was homogenized into the skim milk at 50°F and 2500 lbs. pressure. | 2.0 | 50 | 36 |

The stability of samples B5 and B6, Table 25, shows that the degree of dispersion of the inert material is of importance. When the coagulated casein which was added as an inert phase was dispersed in the skim milk by homogenization, the heat stability of the milk was more than doubled.

Sugars, except glucose in certain concentrations, decrease the heat stability of milk during sterilization. Data showing this effect are given in Table 26. The decreased heat stability may be partly caused by the acid formed from the carbohydrates. However, Whittier and Benton[179] have shown that the addition of sucrose had no apparent effect on acid production in milk by heat. The reactions of the milks in Table 25 were not greatly changed in the presence of sucrose which indicated there was some other reason for the mildly destabilizing effect of this sugar. Dextrose showed a stabilizing influence in the 18 percent solids skim milk and some of the other samples tolerated a higher acidity at coagulation in the presence of dextrose than in its absence. Other experiments with homogenized milks of 18 percent non-fat solids and 8 percent fat showed dextrose heat-stabilized these milks when it was used at 2, 6, and 10 percent levels. In some cases the heat stabilities of the milks containing dextrose were double the values observed for the control samples. Heat stabilization of skim milk was also secured by the addition of dextrin to milks to give final concentrations of

TABLE 26. *The Effect of the Presence of Carbohydrates in Skim Milk during Heating to 248°F upon the Reaction and Heat Stability of the Milk*

Each milk sample was forewarmed to 200°F for 10 minutes, and then concentrated to 30% solids. Dilutions to 6, 12 and 18% solids-not-fat were made with water or carbohydrate solution by weight. (From unpublished data of Webb and Hufnagel[176].)

| Milk Batch Number | Carbohydrate Added | Carbohydrate Concentration,% | 6% S.N.F. | | | 12% S.N.F. | | | 18% S.N.F. | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Reaction | | Heat Stability, min. | Reaction | | Heat Stability, min. | Reaction | | Heat Stability, min. |
| | | | at 14', pH | at coag, pH | | at 14', pH | at coag., pH | | at 14', pH | at coag., pH | |
| 1 | None | | 6.50 | 5.96 | 95 | 6.28 | 6.22 | 27 | 6.13 | 5.96 | 26 |
| 1 | Sucrose | 5 | 6.49 | 5.87 | 100 | 6.28 | 6.25 | 21 | 6.11 | 6.00 | 20 |
| | | 10 | 6.48 | 6.09 | 63 | 6.21 | 6.22 | 19 | 6.10 | 6.08 | 16 |
| 1 | Dextrose | 5 | 6.36 | 6.02 | 44 | 6.11 | 6.09 | 21 | 6.00 | 5.80 | 31 |
| | | 10 | 6.22 | 6.02 | 27 | 6.01 | 6.10 | 13 | 5.90 | 5.98 | 9 |
| 2 | None | | 6.61 | 6.25 | 80 | 6.38 | 6.20 | 25 | 6.20 | 5.98 | 27 |
| 2 | Lactose | 5 | 6.46 | 6.24 | 36 | 6.26 | 6.23 | 13 | 6.09 | 6.08 | 15 |
| | | 10 | 6.39 | 6.21 | 28 | 6.19 | 6.29 | 9 | 6.09 | 6.17 | 9 |
| 2 | Dextrin | 5 | 6.52 | 6.10 | 80 | 6.29 | 6.07 | 43 | 6.10 | 6.25 | 7 |
| | | 10 | 6.51 | 6.10 | 72 | 6.30 | 6.14 | 29 | 6.08 | 6.32 | <0 |

12 and 15 percent solids-not-fat but there was severe destabilization at 18 percent milk solids. Data were obtained on maltose but, since the values were almost the same as those for lactose, the figures have not been included in Table 26.

Dextrose, sucrose and several other sugars have been shown by Ball et al.[10] to inhibit the heat coagulation of egg albumin. The inhibiting influence was exerted on the native protein and was not due to peptization of the coagulated albumin. The effect was accompanied by and considered to be due to the inhibition of sulfhydryl formation by the added sugars.

Some impairment in the amino acids of casein was observed when the casein was refluxed 24 hours at 204°F in 5 percent dextrose solution.[134] This may indicate a decrease in the nutritive value of heat processed foods when both dextrose and casein are present during the processing.

When the ordinary sugars other than dextrose are added to complex milk mixtures a destabalizing influence can be expected. On the other hand when dextrose is used in mixtures containing more than 15 percent milk-solids-not-fat, there should be an increase in protein stability.

The heat stability of milk and cream is affected by the temperature and pressure of homogenization and by the fat and solids-not-fat content. The effect of homogenization temperature upon the heat stability of milks and creams of different fat contents is shown in Figure 17. The skim milk and the concentrated milk of 8 percent fat and 18 percent solids-not-fat are not greatly affected by the homogenization temperature. If creams are heated to the optimum homogenization temperature, 176°F, and are then cooled and homogenized at a lower temperature, such as 140°F, their heat stabilities will be almost, but not quite, as low as if the heating and homogenization were both done at 140°F. The actual temperature of homogenization is more important than the pre-homogenization heat treatment in the case of unconcentrated, high-fat products.

An increase in homogenization pressure causes a decrease in heat stability. But rehomogenization or the use of a second stage valve at a low pressure as shown in Table 27 improves heat stability.

The clumping of fat during homogenization is intimately associated with low heat stability.[44, 46] An increase in the ratio of fat to solids-not-fat decreases clumping in the homogenization process and to a limited extent increases heat stability. However, the improvement in heat stability in this respect soon is overshadowed by the destabilizing effect of the added non-fat solids. The addition of soluble calcium salts has a tendency to increase clumping during homogenization, while sodium citrate and disodium phosphate decrease it.[89] These salts through their effect on fat clumping, indirectly influence heat stability.

**Viscosity and Body.** Body development, though secondary in importance to the attainment of sterility during heat processing, nevertheless is a determining factor in the storage life of canned products. A phase separation of the constituents of a sterilized milk byproduct can be prevented by causing the formation of a highly viscous or gel structure. This structure must not thin excessively during storage. Thinning may allow the formation of a fat layer on top, a precipitation of insoluble curd with heavy particles on the bottom and an objectionable serum or whey in the middle portion of the mixture. Thickeners or binders can be used in those foods which normally contain them, but other products must depend largely upon the milk protein for the production and maintenance of high viscosity.

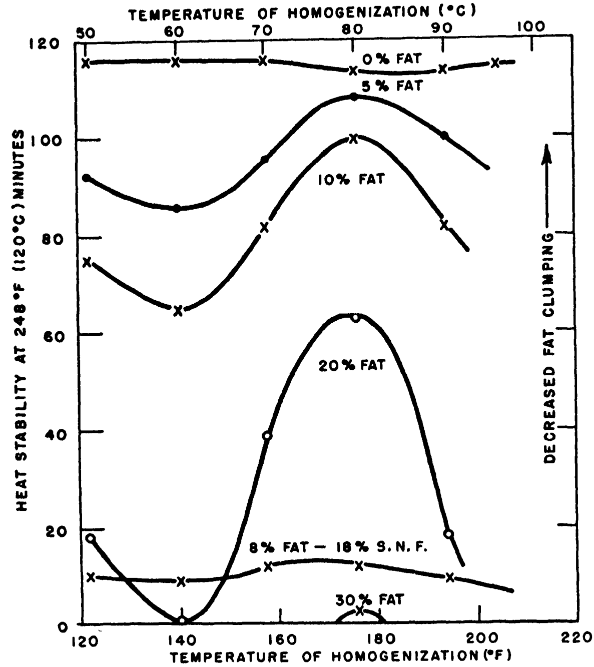

Fig. 17. The effect of the temperature of homogenization upon the heat stability of some milk products. The evaporated milk sample (8% fat, 18% solids-not-fat) was forewarmed to 203°F for 10 minutes, concentrated to 26% solids and then parts of it were heated to and homogenized at the designated temperatures. All other samples, representing normal skim milk, whole milk and cream, received no preliminary treatment other than heating before homogenization. These products contained a percentage of solids-not-fat normal for products of their fat content.

Relative viscosity determinations can easily be made with a MacMichael type viscosimeter. Whitaker and Herrington[181] have developed an equation which makes possible the conversion of degrees retardation as measured with the Mojonnier-Doolittle (torsion pendulum) viscosimeter into centipoises. Potter et al.[136] in a comparative study of torsion pendulum type viscosimeters determined factors for conversion of MacMichael degrees to centipoises. Factors most useful for the determination of the viscosity of sterilized milk byproducts include the following: for wire numbers 24, 26, 28, and 30, the factors are 13.6, 5.8, 1.73, and 0.901, respectively. The factor times the MacMichael reading equals the viscosity in centipoises. These factors are for use with the large plunger at 86°F when the viscosimeter cup is turning at 21 r.p.m.

TABLE 27. *Canned Cream*

Effect of homogenization upon the viscosity, heat stability, and rate of rise of fat in cream of 20.5% milk fat. Temperature of homogenization, 176°F.[167]

| Homogenization Pressure, pounds per square inch | Relative Viscosity at 68°F, before Sterilization (water = 1) | Heat Stability at 248°F, minutes | Distribution of Fat in Can After Two Month's Storage at 73°F | | |
|---|---|---|---|---|---|
| | | | Top Layer, percent | Middle Layer, percent | Lower Layer, percent |
| 0 | 3.75 | 168 | 62.5 | 0.9 | 0.2 |
| 500-0 | 3.80 | 117 | | | |
| 1000-0 | 4.20 | 106 | 51.5 | 7.0 | 0.6 |
| 1000-500 | 4.19 | 110 | 50.0 | 8.0 | 1.4 |
| 1000-1000 | 4.37 | 99 | | | |
| 1000-2000 | 4.82 | 75 | 40.5 | 16.0 | 6.5 |
| 1000-3000 | 5.55 | 46 | 33.0 | 18.5 | 8.4 |
| 2000-0 | 5.12 | 76 | 44.0 | 14.0 | 4.2 |
| 2000-500 | 4.75 | 81 | 43.0 | 15.0 | 5.5 |
| 2000-1000 | 4.87 | 75 | 39.5 | 15.5 | 7.5 |
| 2000-2000 | 5.12 | 55 | 35.0 | 15.5 | 9.0 |
| 2000-3000 | 5.85 | 30 | 31.5 | 19.5 | 11.0 |
| 3000-0 | 7.75 | 38 | 33.5 | 18.5 | 10.0 |
| 3000-500 | 5.50 | 47 | 34.5 | 19.5 | 9.5 |
| 3000-1000 | 5.70 | 43 | 33.5 | 19.0 | 9.5 |
| 3000-2000 | 5.80 | 38 | | | |
| 3000-3000 | 6.62 | 18 | 27.5 | 19.5 | 15.0 |

Eilers et al.[48] in an extensive study of the effect of heat treatments on the viscosities of skim milks of various concentrations found practically no yield value but a true viscosity in those milks that did not approach coagulation during heating. When thickening was due to the imminence of coagulation, a structure was formed and the viscosity measurement was only a relative one. Skim milks that had been concentrated, sterilized and diluted back to their original concentration were considerably more viscous than products sterilized in the diluted condition. Structure developed during sterilization of the concentrate remained after dilution.

In general viscosity measurements of heat processed byproducts can be assumed to represent only relative values. They are not absolute viscosities unless it is shown that there is no yield value or structure. However, relative viscosity values reflect the body the consumer finds and such values are suitable for comparative purposes.

Viscosity is strongly affected by the size and degree of clumping of the fat globules when fat is present. Measurements of fat globule size together with viscosity determinations are used as an indication of storage stability. A convenient method for measuring the fat globules larger than 2 microns in diameter which may be present in homogenized products has been described by Farrall, Walts and Hanson.[50] Doan[43] has made improvements in this method.

The heat processed byproducts of milk may be divided into three groups with respect to viscosity and body characteristics. These groups are (a) the fat-free thin liquids such as skim milk and whey, (b) the evaporated milk group and (c) the cream-soup-type products.

*The thin, fat-free products,* represented by plain or evaporated skim milk and whey or skim milk mixtures, have a viscosity range of 1½ to 30 centipoises. Evaporated skim milk of about 18 percent solids has found some commercial utilization. The coagulation point of the casein is generally not approached in this type of product. However, even the undenatured casein tends to settle to the bottom of the can because its specific gravity is greater than that of the serum. Such separation often occurs during early storage. When the casein reaches the bottom of the can, a denaturation of the protein occurs, due perhaps to concentration effects, with the final formation of a gel structure. Much of the casein in an evaporated skim milk of 18 percent solids is often in the bottom of the can after a year of storage at 86°F. Clear serum then fills the upper half of the tin.

*The evaporated milk type of product* includes modified infant milk, sterilized drinks, and canned cream. The usual range of viscosity of these foods is 20 to 80 centipoises. They may contain fats, sugars, stabilizers and other ingredients which assist the milk protein in development of body.

High fat content in milk and cream is associated with high viscosity values. The increase in the viscosity of cream with increases in butterfat content has been determined by Dahlberg and Hening[39] and by Babcock.[9]

A viscous, well dispersed and adequately protected emulsion must be formed to retard fat separation during storage. To this end the fat is reduced in size by homogenization so that most of the globules are smaller than 2 microns. Calculations on the rate of separation of globules 2 microns in diameter may be made with Stokes' equation for the rate of rise of small spheres at uniform velocity in a viscous medium. Troy and Sharp[164] have shown that fat globules 2 microns in diameter will require 16 days to rise to the top of a quart bottle of milk at 77°F. If appropriate values are used for a milk of 18 percent solids-not-fat, the time which would be required for a 2 micron globule to rise from the bottom to the top of a 14½ ounce can (9.5 cm.) held at 77°F would vary with the viscosity of the milk as follows:

viscosity—10 centipoises—34.5 days
" 20 " 69.0 "
" 30 " 103.6 "
" 60 " 207.2 "

The rate of rise is directly proportional to the viscosity when all other factors are constant. However, at 60°F the viscosity and density of the milk change and approximately 1.6 times as many days are required for the fat to separate to the same extent as it does at 77°F. Most of the globules in well homogenized milks are much smaller than 2 microns, consequently separation is slower than is indicated by the above figures.

The formation of fat clumps was shown by Troy and Sharp[164] to be responsible for the rapid creaming of bottled milk. Clumping occurs in homogenized products when the fat content is high and the temperature of homogenization is low. Leviton and Leighton[112] have shown that the increase in viscosity which usually attends the homogenization of cream and ice cream mixes can be attributed solely to clustering of the fat. The idea is advanced that this increase in viscosity could serve as a basis for the evaluation of an index of the extent of clumping in cream and ice cream mixes. However, changes in protein and salt equilibrium during sterilization seem to preclude the possibility that a clumping index figure determined on processed foods before sterilization would foretell fat separation characteristics during storage. Measurements of fat separation in evaporated milk[174] and in canned cream (Table 27) show that separation is more pronounced in the products of low viscosity.

The casein of milk contributes to viscosity and body development to an increasing extent as the coagulation point of the milk is approached. Deysher et al.[42] showed that the rate of thickening of evaporated milk during sterilization, although variable, became greatest in the 10-minute period preceding coagulation. After coagulation is complete the curd begins to lump and it expresses moisture while the viscosity decreases. The body formed during sterilization becomes greater as the milk solids content of the product is increased.

In the manufacture of evaporated milk it is well known that agitation during sterilization produces a thinner body than is obtained by heating with the sterilizer reel still. The critical period during which agitation causes thinning is toward the end of the sterilization process when the coagulum begins to form but is yet scarcely visible. A product which first shows visible coagulation at 12 minutes while being heated with agitation at 240°F may appear extensively coagulated and lumpy after 18 minutes. However, if agitation is stopped at 12 minutes and heating is continued, a smooth heavy body will be obtained after 18 minutes. The effect of different types of agitation during heating on the viscosity of skim milk is shown in Figure 8, Chapter 5.

Some of the viscosity developed during the pre-coagulation stage is lost in storage. Evaporated milk may lose as much as 40 percent of its original viscosity during the first 10 days of storage at 86°F. The magnitude of the decrease in viscosity is partially dependent upon storage temperature, with temperatures below 60°F strongly retarding viscosity losses. Prevention of the initial storage thinning which the evaporated milk type of products undergo would greatly improve the storage life of these foods. The separation of fat and in some cases even the precipitation of casein could then be retarded. But the mechanisms of the changes which the heat-treated casein complex undergoes during sterilization and aging are not understood. There appears to be a dehydration of the complex followed by denaturation and precipitation which finally may result in the formation of a layer of insoluble protein in the bottom of the can.

*Cream-soup-type products* are often a semi-gel and may range in viscosity from 100 to 800 centipoises or more. Whereas the evaporated-milk type products possess only a slight structure, viscosity values on cream soups decrease with stirring or beating. Such viscosity values should be considered as relative rather than absolute unless plasticity determinations are also made. Well emulsified fat contributes to body development in this class of foods just as it does in the evaporated milk type of product.

Some of the factors which affect the viscosity and body of cream-style foods have been studied.[175] The presence of ingredients foreign to milk such as vegetable constituents, salt, starch and meat products causes the coagulation of the milk protein in the early stages of sterilization. The pre-coagulation increase in viscosity ceases long before sterilization is complete. Starch or flour binders must therefore be employed to take up the moisture expressed by the coagulating casein. A soft viscous structure is formed by the starch-casein gel which gives the cream-style foods their characteristic body. Finely divided inert or pulpy material such as can be produced by the homogenization of vegetable cellular structures, encourages the development of a smooth coagulum.

Agitation during sterilization of most cream-style products should be avoided. The starch-casein gel which begins to form in the early stages of processing will break up and express water if it is unduly disturbed. This produces a coarse, coagulated type of body which presents an unattractive appearance and permits separation of constituents during storage.

**Color and Flavor.** Brown color and cooked flavor are closely associated with each other in heated milk products and both increase with increases in time and temperature of heating. Color production is generally attributed to caramelization of the lactose in the presence of milk protein. The flavor of heated milk is associated with the liberation of sulfides and the formation of sulfhydryl groups. Townley and Gould[159] observed that when milk was heated at 203°F the early, rapid liberation of sulfides decreased to zero after 210 minutes while the cooked flavor increased during the first 30 minutes after which it became more of a caramel flavor. The first brown color appeared after 90 minutes when the caramel flavor became quite distinct and sulfide liberation was rapidly subsiding. A lag observed in the progressive darkening in the color of milk during sterilization occurred simultaneously with a marked decrease in labile sulfur liberation.[128, 159]

Tarassuk found a decrease in the oxygen and an increase in the carbon dioxide of the free space in cans of evaporated milk after sterilization.[156] It was suggested that the flavor and color of canned milk could be improved by lowering the oxygen available in the can before the milk was sterilized.

*Color* formation in milk was first attributed by Orla-Jensen and Plattner[132] to a reaction which involved both the lactose and the casein of the milk. Other workers[138, 168] found that color was formed in heated solutions of lactose in the presence of amino acids or milk proteins. Wright[185] considered the color to be due to a caramelization of the lactose which was catalyzed by calcium caseinate and that the casein in adsorbing the pigment remained unchanged. A study of the chemical factors involved in the browning of heated milk was made by Kass and Palmer.[106] They found the discoloration to be accompanied by the development of acidity, a pronounced fall of optical activity, a comparatively slight loss of copper-reducing ability and a conversion of lactose to ketoses or substances not oxidized by sodium hypoiodite. Kass and Palmer concluded that the origin and behavior of the color could be explained on the basis of the caramelization of lactose by the casein and the adsorption of the lactocaramel by the colloidal caseinates. Some characteristics of the browning reaction as recently summarized by Kass[105] have been given in Chapter 5.

The color of evaporated milks made under various conditions has been measured and expressed in numerical terms according to the Munsell system[19, 172] and by spectrophotometric measurement of reflectance.[128] An increase in heat during manufacture produces an increase in color, the change being of a catalytic nature. The color developed during sterilization affects lightness, chroma and hue, while during storage the color deepens because of an increase in chroma only. Milks may be forewarmed by high-temperature short-time methods to give them greater than normal heat stability without adding to the color produced by the usual forewarming to 203°F for 10 minutes. The color developed in evaporated milks during heating is not proportional to the solids content of the milk. A milk of 26 percent solids will be lighter in color after sterilization than a milk processed at 32 percent solids and subsequently diluted back to 26 percent solids.

A grayish-green discoloration appears in coffee to which evaporated milk containing 3 to 5 parts per million of iron is added. The iron in the milk reacts with the tannins in the coffee to produce this undesirable condition. These facts were established in recent studies by Gould[64] and by Cole and Tarassuk.[31]

*Flavor.* Gould and Sommer[65] first showed that the cooked flavor of milk was characterized by the formation of sulfides in the milk when it was heated above 162 to 169°F. Their report includes a review of previous work. More recently there have been other important contributions to the knowledge of heat-produced flavors in milk. These have included methods for quantitatively determining sulfides,[29, 36, 84, 157] and volatile acidity,[121] the establishment of the whey protein and the fat adsorption membrane as the chief source of heat labile sulfides,[104, 158] and studies on the effect of various substances and processing methods on sulfide formation.[61, 159]

The sulfides are active antioxidants and appear to be responsible for preventing the development of oxidized flavors in evaporated milk type products. Musher has used caramelized milk solids, particularly heat treated whey solids, as an antioxidant in food preparations.[124, 125, 126]

The sulfur compounds of milk are apparently involved in the activated or burnt flavor which develops under certain conditions during ultraviolet irradiation of milk.[53, 54]

Not all the substances that form during the heating of milk have been isolated and it is difficult to evaluate the contributions of the known compounds to cooked flavor. The prominent role of sulfides in flavor formation is well known. Lactic acid usually constitutes only 5 percent to 7 percent of the total acidity produced in milk by heat. Formic acid constitutes about 84 percent of the volatile acidity and about 57 percent of the total acidity in skim milk heated at 241°F for 2 hours.[60] Furfuryl alcohol has recently been isolated from skim milk heated to 260°F for 90 minutes.[135]

## METHODS OF MANUFACTURE

Production methods and formulas for the preparation of a number of packaged and sterilized byproducts of milk will be presented. Some of these foods are commercial preparations, the others are laboratory products which it is hoped will stimulate the development of new uses for milk byproducts. The directions include sterilization processes for the foods when they are sealed in No. 1 (200 x 400) cans. If other can sizes are used, suitable time and temperature adjustments should be made. Sterilization of the foods in sealed containers is unnecessary if they are to be consumed at once. If the sterilization processes are not applied, many body and flavor difficulties will not be met. In some cases the formulas may be adjusted to suit the modified processing conditions.

A good chef often has several recipes for a favorite dish. Similarly several formulas could be given for the products discussed here. A single formula for each product has been recommended but plant chemists are urged to make whatever modifications may be found necessary for their own operating conditions.

**Canned Cream.** *Manufacturing Processes.* Following the great development of evaporated milk during World War I, interest was shown in the production of canned cream. The next decade brought limited commercial activity which was based upon the discovery of several processes for cream canning. But manufacturers are still seeking a process which will produce an entirely satisfactory product.

Canned whey cream is discussed later in this chapter under *Canned Whey Products.*

Fenlon[51, 52] patented a process for canning cream of 18 to 24 percent fat which consisted of increasing the milk-solids-not-fat of the cream, either by concentration or by the addition of concentrated milk. The high milk-solids content produced a heavy bodied product which retarded fat separation during storage. Fenlon first condensed 18 percent fat cream to a total solids content of 32 percent but later modified this procedure by raising the solids-not-fat content to not more than 10 percent by the addition of condensed or dry skim or whole milk. The cream of 18 percent fat and 7 percent to 10 percent milk solids-not-fat was then heated to 140 to 170°F, homogenized two or more times at 2000 pounds per square inch pressure, canned and sterilized at temperatures between 179 and 250°F. This process was subsequently modified by Frohring[55, 56, 57] whereby 1 percent sodium caseinate was added to a high fat cream, the cream then being diluted to 18 percent fat with skim milk, homogenized and sterilized. The sodium caseinate increased the heat stability of the cream. In place of adding sodium caseinate, Germann[59] raised the casein content of a cream by homogenizing the milk before separation. The formation of new fat surfaces increased the amount of protein adsorbed on the fat.

Early British descriptions of cream canning were principally concerned with the use of conventional processes and equipment.[113, 147]

The precise relationships which exist between the temperature and pressure of homogenization of creams of different fat contents and their viscosities and heat stabilities have been discussed earlier in this chapter. A simple process for canning cream consists in heating 20 percent fat cream to 176°F and homogenizing it at 3000 pounds pressure, sealing it in tight containers and sterilizing it at 245°F for 12 minutes.[166, 169]

The Grindrod process[68-76, 78-81] for sterilizing liquid foods has been made the basis for commercial production of sterilized cream.[88] Approximately 0.10 percent to 0.25 percent by weight of sodium alginate is added to fresh cream of 18 percent fat and 0.15 percent titratable acidity or to cream of 30 percent fat and 0.14 percent acidity. The cream is preheated under pressure in closed hotwells and then subjected to the sterilizing effect of high velocity, high temperature steam jets. Sterilization is attained by using either 260°F for 3 minutes, 270°F for 0.65 minute or 280°F for 0.22 minute. The Grindrod patents provide for removal of the condensed vapor under vacuum and simultaneously cooling the cream to the homogenization temperature of not less than 150°F. Homogenization pressures of 2000 pounds, first stage, and 500 pounds, second stage, are suitable for light cream but the temperature and pressure are gaged to produce the proper viscosity in the cream. After homogenization the cream is cooled and held for bottling or canning. All operations subsequent to sterilization are performed under aseptic conditions with elaborate use of conditioned air and germicidal lamps. The 30 percent whipping cream can receive little or no homogenization and must be shipped under refrigeration to prevent it from churning.

If the packaging equipment is not sterile, Grindrod advocates resterilization of the canned product in an autoclave at 212 to 230°F for 10 minutes.[81] A process for sterilizing cream by pumping it through a high temperature heater has been patented by Grasse[66, 67] but no provision is made for sterile packaging.

The temperature and pressure used in the homogenization of cream control the heat stability and body of the finished product. Maximum heat stability but minimum viscosity will result when the cream is homogenized at 176°F.[171] Conversely cream of semi-solid consistency such as is packaged in Denmark and Great Britain can be produced by homogenizing at 110 to 140°F.[113] It should be noted that when homogenization is performed after sterilization,[88] the temperature and pressure of homogenization need be adjusted only to produce a cream of the desired viscosity and with stability against feathering in coffee. The manufacturer is thus relieved from consideration of the effects of homogenization upon stability in the sterilizer but he must solve the problem of aseptic packaging.

*Storage of canned cream.* The commercial development of canned cream has been retarded by the fat separation which generally develops during storage. A high viscosity which can be secured by using low temperatures and high pressures of homogenization will retard fat separation. The effects of homogenization and rehomogenization upon viscosity, heat stability and fat separation are shown in Table 27.[167] Canned cream which is forewarmed and homogenized so that sterilization will cause an incipient coagulation and a semi-gel structure, will not show fat separation. However, if the coagulum develops too far, wheying off in storage will occur. In the United States where a free-pouring, fluid product is desired, vegetable stabilizers up to 0.4 percent have been used to retard fat separation.

Jackson, Howat and Hoar[101, 102] studied the purplish discoloration and the corrosion or pitting of the tin which was found in samples of canned cream. The trouble was caused by the action on the tin of liberated sulfur compounds. It was suggested that the defect could be prevented by using (a) only cream from good milk, (b) sound well-tinned cans, (c) a minimum of stabilizer, (d) the lowest heat treatment which would give a sterile product, and (e) a short storage period.

Strains of *B. subtilis* with spores capable of withstanding temperatures up to 248°F for 40 minutes have been isolated from commercial canned cream. These organisms caused bitterness and thinning of the cream.[129]

Horner[93] observed an increase in the tin content of canned cream from 12 p.p.m. in the fresh product to 73 p.p.m. after about one year storage. He found a similar increase in evaporated milk and about half this increase in sweetened condensed milk.

**Ice Cream Mix.** It is possible to sterilize ice cream mix in cans but a noticeable cooked flavor develops and there are limitations in composition which must be observed. The high heat treatment to which sterilized mixes are subjected gives them improved whipping properties. This improvement is somewhat in proportion to the severity of the heat treatment. Unless a semi-solid body is developed during sterilization, fat separation in storage will occur because of the low ratio of milk-solids-not-fat to fat and the high density of the aqueous phase of the mix.

Corbett and Tracy[33] found that by careful processing an ice cream mix of 36 percent total solids, including 9 percent milk-solids-not-fat, could be sterilized. Stabilizers and egg yolk slightly lowered the heat stability of the mix. Homogenization was carried out at 2000 to 2500 pounds pressure and the use of a second stage valve set at 500 pounds pressure improved heat stability. Other experimental work[176] has shown that an increase in heat stability may also be obtained by forewarming the skim milk of the mix to 203°F (95°C) for 10 minutes before condensing it, by keeping the sugar and milk-solids-not-fat content of the mix as low as the quality standard will permit, and by homogenizing the mix at 176°F.

The following process may be formulated for the preparation of a canned sterilized ice cream mix. Use sweet, high quality, heat stable milk and cream. Forewarm the skim milk to 203°F for 10 minutes before it is condensed or dried. Make up a mix of 12 percent fat, 9 percent, or if sufficiently heat stable, 10 percent milk-solids-not-fat, 12 percent to 13 percent sugar and 0.3 percent stabilizer. Preheat the complete mix to 176°F and homogenize it at 2000 to 2500 pounds pressure on the first valve and 500 pounds on the second. Can the mix and sterilize it at 245°F for 15 minutes. A trace of disodium phosphate may be used to improve heat stability. When a batch sterilizer is employed, stopping the reel during the last 5 to 8 minutes will assist in producing a smooth heavy body, especially when the coagulation point of the mix is being approached.

In view of the difficulties involved in the production of a satisfactory sterilized mix and the recent successful preparation of dry mixes, the future of canned, sterilized fluid mix is not promising.

Attempts have been made to prepare sterilized and aseptically packaged mixes but the success of this type of operation must depend upon the available aseptic packaging facilities. Developments in this field were discussed earlier in this chapter.

**Canned Milks.** Processes will be discussed for the canning of five types of milk: skim milk, evaporated skim milk, modified milk for infants, flavored milk drinks, and vitamin D concentrates in milk. A complete discussion of the manufacture of evaporated milk, the usual form of canned milk, may be found in Hunziker's text.[97] The same basic processes and the same equipment are used in the manufacture of evaporated milk and in the preparation of the special canned milks which will be discussed here.

*Sterilized skim milk* is not and probably never will be an article of commerce. It is used as media by bacteriologists and studies of its storage stability may be helpful in the development of a stable sterilized whole milk. Skim milk may be canned and sterilized at 240°F for 15 minutes. Coagulation will not occur in normal sweet skim milk.

The protein of sterilized skim milk settles rapidly during storage. Complete protein separation, which may occur in a few months at 86°F, apparently is due to the very low viscosity of the skim milk. When fat is present, especially when it is homogenized into the skim milk, or when whole milk is used, protein separation is retarded. If a perfect balance could be attained by causing all the casein of milk to be adsorbed on very small fat globules, separation might be prevented in sterilized whole milk independently of its viscosity.

With the advent of fast, high-temperature sterilization and aseptic packaging techniques there has been much interest in the processing of surplus milk from high producing areas. It is anticipated that such a process would result in cheaper milk for the consumer.[144] However, changes in the fat and protein dispersion occur more rapidly in the quickly sterilized, aseptically packaged milks than they do in milks sterilized by the old conventional methods. Aseptic packaging techniques produce milk with only slightly more cooked flavor than pasteurized milk, but additional research is needed to devise methods for retarding phase separation during storage.

A sterilized bottled milk is being produced in Great Britain.[1, 2, 90] This product is handled in fluid milk channels and is not held long in storage.

Whole milk has been canned and heated at 240°F for 2 hours to produce a special hypo-allergic milk.[58] This product has a light chocolate color, produces a fine curd when acidified and is said to be useful in special diets.

*Evaporated skim milk.* The production of 16,844,000 pounds of evaporated skim milk as case goods, in 1947, was small in comparison to the three billion pounds of evaporated whole milk which was manufactured. Evaporated skim milk is made principally for export. According to the definition and standard of identity of the Food and Drug Administration it must contain not less than 20 percent of milk solids.

Small quantities of evaporated skim milk to which vegetable oils and vitamins A and D are added, have been made according to a patented process.[87] This product is a filled milk and is not to be confused with normal evaporated cows' milk.

Fresh skim milk which has not been subjected to changes caused by bacterial growth should be used in the manufacture of evaporated skim milk. The milk is forewarmed at 195 to 210°F for about 10 minutes to give it adequate heat stability. It is evaporated to 20 to 22 percent solids content, canned and sterilized at 240 to 242°F for 15 minutes. Calcium chloride or disodium phosphate may be used for stabilization if necessary. The data plotted in Figure 15 shows that if the solids content of a skim milk exceeds 22 or 23 percent it may not withstand the heat treatment necessary to sterilize it. Evaporated skim milk, being almost fat-free has a viscosity of 7 to 30 centipoises. The protein of evaporated skim milk separates during storage to a greater extent than the protein of evaporated whole milk. An evaporated skim milk of 18 percent solids content and a viscosity of 7.2 centipoises showed a protein separation after storage at 86°F for 104, 211 and 365 days of 15 percent, 39 percent, and 86 percent, respectively, of the total protein in the milk.[176] Separation at lower storage temperatures or in milks of higher viscosity would probably occur more slowly.

*Modified Milk for Infants.* Milk may be modified to a composition suitable for the special needs of infants. The modification usually consists in the addition of a carbohydrate such as lactose to a milk or partially skim milk base. Sometimes part of the milk fat is replaced with vegetable fat to aid digestibility by certain infants. The composition of modified milk generally approximates that of human milk. A summary of the comparative values of vitamins and other nutritional factors of cows' milk, as well as human milk is available.[109]

Modified milks are manufactured in relatively small quantities in a few evaporated milk plants in the United States. The composition of whole, evaporated and modified cow's milk, and of human milk is shown in Table 28. Modified milk No. 1 is of the same basic composition as normal evaporated milk but it is heavily fortified with vitamins and minerals. One 14½ ounce can contains vitamins A—2000 U.S.P. units, $B_1$—1.0 mgm., C—50 mgm., D—500 U.S.P. units, riboflavin—3.0 mgm., niacin—5.0 mgm. Each can of milk No. 1 also contains essential minerals but it is intended that carbohydrate be added to suit individual needs. It is claimed that the vitamin C of this milk has been successfully stabilized and that the vitamin potency is retained in storage.[117] Modified milks No. 2 and No. 3, when diluted with equal parts of water approximate the composition of human milk except for the protein in No. 3.

TABLE 28. *Composition of Milks Used for Infant Feeding*

The evaporated and modified cow's milks are diluted before feeding, usually to twice their volume.

| Kind of Milk and Sample No. | Water, Percent | Protein, Percent | Fat, Percent | Carbohydrate, Percent | Ash, Percent |
|---|---|---|---|---|---|
| Human milk[1] | | | | | |
| normal | 87.55 | 1.29 | 4.06 | 6.88 | 0.20 |
| concentrated | 74.00 | 2.70 | 8.52 | 14.36 | 0.42 |
| Cow's milk | | | | | |
| whole | 87.90 | 3.13 | 3.65 | 4.50 | 0.72 |
| evaporated | 74.00 | 6.78 | 7.91 | 9.75 | 1.56 |
| Modified cow's milk[2] | | | | | |
| No. 1 | 74.00 | 6.80 | 7.90 | 9.00 | |
| No. 2 | 75.00 | 3.75 | 6.25 | 14.25 | 0.75 |
| No. 3 | 68.00 | 6.60 | 6.60 | 17.40 | 1.40 |

[1] Values for normal human milk averaged from figures given in Fundamentals of Dairy Science.[6] Concentrated human milk calculated for compairson on a basis of 74% water.

[2] Composition of typical commercial modified milks as stated on the can labels.

The process of manufacture of the modified milks is almost identical with the process of manufacture of evaporated milk. The principles discussed early in this chapter should be applied to each manufacturing step. The milk is first standardized with respect to fat and protein content. It is then forewarmed to a high enough temperature to give the finished product adequate heat stability. A temperature of 200 to 210°F for 15 minutes should generally be satisfactory. If extra carbohydrate is used it should be dissolved in water and heated to about 170°F, then drawn into the vacuum pan after the milk. The batch is evaporated to the required concentration, canned and sterilized. Modified milk often has a greater viscosity than evaporated milk and therefore it may show less fat separation on storage.

A low protein product, such as modified milk No. 2, Table 28, is concentrated only slightly. Such a product could be compounded from plain and concentrated whole milk, cream and a carbohydrate without drawing the complete mixture into the vacuum pan. This mixture should be heated to 160 to 180°F, homogenized, cooled to filling temperature (60°F) canned and sterilized.

The choice of carbohydrate to be added to modified milk is governed by nutritional and economic considerations. Dextrose or a blend of dextrose and sucrose, of dextrose and maltose or of dextrose and lactose may be used. Dextrose is a low cost sugar which is high in nutritive value. The data of Table 26 shows that most carbohydrates decrease the heat stability of skim milk, but that 5 percent dextrose slightly increases the heat stability of the 18 percent solids skim milk. Fresh, sweet whey is an excellent source of lactose but care must be taken not to allow the whey protein and salts to adversely affect the heat stability of the milk. Meyenberg[119] has advocated boiling the whey and other ingredients separately to attain adequate heat stability in the finished product. The milk and cream are boiled in one hotwell, the whey in another, and the carbohydrate solution (dextrose) in a third hotwell. The mixtures are drawn into the vacuum pan successively.

*Sterilized flavored milk drinks* have been manufactured on a limited scale by a few processors. They have not been widely accepted, possibly for economic reasons but more probably because they cannot yet be made equal in flavor and body characteristics to a freshly prepared drink. Ready-to-drink beverages may be sealed and sterilized in bottles or cans. The bottled products are sometimes distributed through soft drink channels. During World War II, the evaporated milk industry made experimental packs of flavored milks which were desired by the Army as "morale builders." A chocolate milk and a caramel milk, both ready to drink directly from the can were the most promising products which were developed.

A ready-to-drink flavored milk should contain 16 to 24 percent total solids. The milk fat content may vary between 1 and 5 percent. The product must be heavy enough to retard excessive rising of fat or settling of flavoring ingredients, but thin enough to be palatable as a beverage. The viscosity may vary between 10 and 60 centipoises. Since there will be some storage separation, cans should be shaken before they are opened. Corn starch or a mixture of sodium alginate and Irish moss are among the best stabilizers to use. The algin-moss mixture lowers the heat stability of milk more than does corn starch.

Sterilized chocolate milk (not concentrated) may be prepared as follows. To a fresh milk of 1 percent milk fat add 6 percent by weight of sugar and 1.8 percent good quality cocoa. Heat the mixture to 160°F, and homogenize at 2500 pounds pressure. Mix 0.2 percent corn starch with some of the milk, then stir this mixture into the batch, heating the product with stirring to 176°F. The chocolate milk may be filled directly into the containers or cooled to a lower filling temperature to facilitate the filling operation. Sterilization of 14½ ounce cans can be carried out at 240°F for 20 minutes. When an algin-moss mixture is used as a stabilizer, it should first be mixed with the sugar and then added to the milk at a temperature of 160°F. The complete mixture is then homogenized.

A process for the manufacture of a sterile chocolate beverage using agar as a stabilizer and direct steam injection as a means of sterilizing the milk has been described by Grindrod.[77]

Chocolate milk is sometimes sterilized in bottles rather than in cans. The agitating sterilizer can be built to accommodate bottles. The pressure inside and outside of the bottle must be balanced during processing so that the caps will not be forced off the bottles. A study has been made of the relationship between the internal and external pressure on the bottle and the time and temperature of heating as applied to chocolate milk.[4]

A method for bactericidally pretreating the cocoa ingredient before it is mixed with the milk has been devised.[99]

Caramel milk is made by mixing a caramel base with a partly concentrated milk, then canning and sterilizing the product. Caramel flavor cannot be developed simply by sterilizing a mixture of milk and sugar. During continued heating of such a mixture in a sealed container, a predominantly cooked flavor is produced which should not be confused with true caramel. Caramel flavor develops when milk protein and the sugars, generally sucrose and dextrose, are heated and concentrated together at high temperatures. The heat treatment carries the milk protein beyond the coagulation point and constant agitation is necessary to obtain a smooth blend of all ingredients. A formula for a caramel flavor base suitable for the preparation of caramel milk or for other uses such as topping for desserts is presented in Table 29. Caramel milk should contain 14 pounds of the caramel base of Table 29 in each 100 pounds of finished milk. The formula and composition for caramel milk is given in Table 30. The 86 pounds of milk may be added as milk of 3.84 percent fat and 9.3 percent solids-not-fat. Such milk should be forewarmed to 203°F for 10 minutes with the caramel base and homogenized at 2500 pounds pressure, cooled for filling cans and sterilized at 240°F for 20 minutes. An experimental batch of caramel milk can be easily prepared by mixing 14 pounds of caramel base with 42½ pounds of water and 43½ pounds of unsterilized 26 percent evaporated milk. The mixture should be warmed to 110°F, homogenized at 2500 pounds pressure, cooled to about 60°F to fill the cans and sterilized at 240°F for 20 minutes.

TABLE 29. *Formula for Caramel Flavor Base*

| Product | Wt. of Product, lbs. | Weights of Ingredients in Each Product | | |
|---|---|---|---|---|
| | | Fat, lbs. | Solids-not-fat, lbs. | Total Solids, lbs. |
| Cream (30% fat) | 33.3 | 10 | 2.1 | 12.1 |
| Condensed skim milk (30% solids) | 15.0 | — | 4.5 | 4.5 |
| Corn sirup | 45.0 | — | — | 36.0 |
| Sucrose | 14.0 | — | — | 14.0 |
| Salt | 1.0 | — | — | 1.0 |
| Totals | 108.3 | 10 | 6.6 | 67.6 |

Heat the complete mixture with stirring to 160°F, and homogenize it at 2500 lbs. pressure. Cook in a candy kettle with double action stirrer to 238°F, cool slightly and add 8¼ lbs. of water and vanilla flavoring to suit the taste. Stir well and pour into a suitable receiver. Approximately 84 pounds of base containing 20% water will be recovered.

TABLE 30. *Formula for Caramel Milk*

| Product | Weight of Product, lbs. | Weights of Ingredients in Each Product | | | | |
|---|---|---|---|---|---|---|
| | | Fat, lbs. | Milk-solids-not-fat, lbs. | Sucrose, lbs. | Corn Sirup Solids, lbs. | Total Solids, lbs. |
| Caramel base (Table 29) | 14 | 1.7 | 1.1 | 2.3 | 6.0 | 11.1 |
| Milk | 86 | 3.3 | 8.0 | — | — | 11.3 |
| Totals | 100 | 5.0 | 9.1 | 2.3 | 6.0 | 22.4 |

Observations[176] on the storage characteristics of caramel milk show there was a tendency toward a settling of caramel in milks which were below about 35 centipoises in viscosity or in samples made from caramel base containing vegetable fat in place of milk fat. The decrease in viscosity during the first days of storage which is characteristic of evaporated milk was not observed in caramel milk. Many samples of caramel milk did not change in viscosity during storage at 86°F for one year.

Concentrated flavored milks which require dilution with an equal part of water are more difficult to process than the dilute preparations. But they are economical in their space and container requirements and their heavy body offers protection against separation during storage. The solids content of a concentrated drink should be 26 to 32 percent. The viscosity will range between 60 and 300 centipoises, the body in some cases being a semi-gel. An attractive high solids chocolate milk may be made of the following composition: total milk solids 18 percent, sugar 8.3 percent, cocoa 1.4 percent, corn starch 0.3 percent. The ratio of fat to milk-solids-not-fat in this case should be the same as for evaporated milk, 1:2.27. If skim milk is used the milk solids content should be somewhat lower. Vanilla may be added to suit the taste. The raw milk should be forewarmed to 203°F for 10 minutes or to higher temperatures if this does not give enough heat stability. The milk is next evaporated to 18 to 20 percent solids, the cocoa and sugar added and the mixture homogenized at 2500 pounds pressure. The starch is stirred in, the batch standardized, canned and sterilized at 240°F for 20 minutes. A minimum of agitation during sterilization will generally be desirable to prevent the development of a firm coagulum. In a batch sterilizer the reel should be held still during the last half of the holding period.

*Vitamin D concentrates* in evaporated milk are special preparations of the vitamin dispersed in evaporated milk and sterilized. The vitamin D potency of the product may vary but it is high enough so that a small can of the concentrate will fortify one or more large vats of milk. One such preparation contains 20,000 U.S.P. units per gram of milk. The evaporated milk type of concentrate is used almost exclusively for the fortification of market milk. It has several advantages over the other type of concentrate, a solution of vitamin D in a vegetable oil. The evaporated milk concentrate is a sterile product in a carrier consisting of milk products which will disperse readily in fluid milk. The evaporated milk concentrate is easy to store and easy to measure and add to the product being fortified.

Vitamin D concentrates in evaporated milk are manufactured from grade A milk.[27, 149, 150] Activated ergosterol is first dissolved in pure butterfat and the solution is assayed. The fat concentrate is dispersed in evaporated milk, the mixture is homogenized, canned and sterilized according to the usual evaporated milk procedure. The finished product is again assayed to establish the fact that the declared amount of vitamin D is contained in the product. The direct homogenization of a cod-liver concentrate into either cream or evaporated milk, followed by canning and sterilization has been practiced.[23]

A sterilized milk product such as normal evaporated milk, which is not intended for use as a vitamin concentrate, may be fortified directly by homogenizing an oil concentrate into the milk.[7] The oil concentrate is produced by irradiating a sterol of animal origin with ultraviolet light to attain a vitamin $D_3$ concentration of 400,000 U.S.P. units per gram of oil.

**Canned Cream-Style Products.** *Cream-style soups* are manufactured commercially in a variety of flavors and of different compositions. Modifications of the soups are sometimes prepared with meat and vegetables or with eggs and cereals. These foods are strained or homogenized and then canned and used for infants, invalids or persons on special diets. Coagulation of the milk protein in the products is unavoidable but it is important to control the kind of curd which forms. This may be done by the use of the optimum quantity of starch or flour binder to take up moisture expressed by the casein, by the comminution of inert pulp and fibrous material by homogenization, by sterilization of the soup with a minimum of agitation, and by the use of milk products which have maximum heat-stability characteristics.[175]

TABLE 31. *Formulas for Whey and Milk Soups*

| Type of Soup | Ingredients | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Dried Whey, % | Dried Skim Milk, % | Milk Fat, % | Flour or/and Starch, % | Salt, % | Sugar, % | Fresh Vegetable, % | Added Water, % |
| Whey soups | | | | | | | | |
| Celery | 5.0 | | 4.0 | 3.0 | 1.0 | | 16.0 | 71.0 |
| Tomato | 4.0 | | 4.0 | 2.8 | 1.0 | 1.0 | 70.0 | 17.2 |
| Pea | 3.5 | | 4.0 | 2.0 | 1.0 | | 50.0 | 39.5 |
| Milk soups | | | | | | | | |
| Mushroom | | 4.0 | 4.0 | 2.5 | 0.7 | | 8.0 | 80.8 |
| Tomato | | 4.0 | 4.0 | 3.0 | 1.0 | 1.0 | 70.0 | 17.0 |
| Pea | | 3.5 | 4.0 | 2.5 | 1.0 | | 50.0 | 39.0 |

Blanch vegetable and cut or mash it. Vegetables may be cooked in some of the water used in the formulas if it is found necessary to do so. Mix juice, cooking water and other finely dispersed ingredients, withholding any pulpy material which it may not be desirable to homogenize. Warm the mixture to 110°F and homogenize it at 2500 pounds pressure. Add pulp and flour or starch binder if they were not added before homogenization. Heat the complete soup, with stirring, to 176°F, can, and sterilize at 240°F for 60 minutes. A holding time of 30 minutes may be used for tomato soup. Agitation of the cans in a revolving cage during sterilization should be avoided.

Typical formulas for milk or cream-style soups are presented in Table 31. In general, a satisfactory formula should include 3 to 4 percent milk-solids-not-fat, 2 to 4 percent milk fat, 2 to 3 percent starch or flour, about 1 percent salt and the remainder vegetable extract, vegetable pulp and soup stock.

Every ingredient added to a cream soup exerts an influence on the kind of body which is produced during processing. The solution of difficulties lies in making experimental packs and in varying the soup constituents, bearing in mind the principles outlined early in this chapter.

*White sauce*[175] which will withstand storage at room temperature for several years with little change can be prepared by mixing 94 percent whole milk by weight, 5 percent flour and 1 percent salt. The mix should be heated to 185°F with constant stirring, canned and sterilized at 240°F for 25 minutes. The gel structure which is built up by the casein and the flour is relatively permanent and does not permit any appreciable expulsion of whey or separation of fat during storage. Towson and Trout[162] found that white sauces made from homogenized milk showed a separation of fat in constrast to those made from unhomogenized milk which appeared smooth. These sauces were not canned and sterilized. Canned white sauce has recently become an article of commerce.[3] Because of its stability of body and flavor it should merit consideration by both packers and consumers.

*Corn starch dessert* is a product similar to white sauce. It may be made by mixing 86 percent whole milk, 10 percent sugar, 4 percent corn starch and vanilla to flavor, heating the mix to 185°F, while stirring, then canning and sterilizing it at 240°F for 35 minutes. Corn starch desserts are easily prepared in the home but the canned product has commercial significance because it illustrates again the type of gel preparation which can be so readily produced with milk casein and a suitable binder. A custard type pudding can be made by the addition of eggs to a modified corn starch dessert formula. Studies on the use of milk in custard type products which were not canned have been made.[28, 91]

**Canned Whey Products.** Sweet, separated cheese whey is a byproduct rich in nutritive value but poor in taste appeal. When it is combined with other foods it is limited in its ability to improve the flavor and attractiveness of the new product. The high lactose and salt content of whey solids can give rise to solubility difficulties and to the production of a salty taste.

Whey protein is a very useful constituent in certain heat-processed whey foods. It coagulates as a fine, flocculent, soft, milky white precipitate which mixes well with the other suspended materials of the food. The coagulum is not tough and lumpy as is the case with a casein curd. Highly acid foods, when mixed with whey, do not significantly change the characteristics of the whey-protein curd which forms during heating. These properties of whey protein, together with the food value, milk flavor and comparatively low cost of whey solids, form the basis for their utilization in sterilized food preparations.

*Whey cream* is the product which is obtained by the separation of the fat from cheese whey. The low protein content of whey cream causes it to whip and churn quickly. Homogenization sometimes destabilizes the emulsion toward heat and causes the fat to oil off. This instability together with the fact that sterilized whey cream rapidly develops an oxidized flavor indicates that the fat globules in the product are inadequately protected.

The possibilities of canning whey cream have been investigated.[176] There is no heat stability problem and only a slight color appears when whey cream is sterilized, but during storage there is rapid fat separation as well as the development of an oxidized flavor. The appearance of these defects was postponed from several days to several weeks by separating the whey to obtain a cream of 10 percent fat, heating the cream to 190°F for 5 minutes, cooling it to 95°F, and reseparating it to produce a cream of 60 percent fat content. By this procedure the whey protein content of the cream was increased approximately 100 percent. The 60 percent cream, after canning and sterilization, was smooth and heavy. It could be diluted with an equal part of milk and easily whipped. A canned whey cream of 60 percent fat might have commercial possibilities if it contained an antioxidant and sufficient whey protein to adequately cover the fat.

*Whey drinks* prepared from whey and a fruit or vegetable juice are high in nutritive value but often inferior in flavor to the plain juice without whey. Tentative experimental processes for a prune and a tomato beverage will be described.[176] They are of interest because they illustrate the type of beverage which can be prepared and because the brief descriptive process may stimulate the development of improved products.

Prune juice can be made by extracting dried prunes with a clarified whey. Fresh cheese whey should be allowed to develop acid until pH 4.8 is reached. The whey is further acidified with citric acid or preferably lemon juice to pH 4.5, heated to boiling and the clarified liquid decanted or filtered off. One part by weight of dried prunes is extracted with 2 parts of the clarified whey either by cooking the prunes and whey together and filtering off the extract or by leaching the prunes with successive quantities of the whey. The clear extract is canned and sterilized at 212°F for 30 minutes. The reaction of the finished juice will be about pH 3.8 to 4.0. It is of interest to note that cans of plain prune juice prepared by water extraction of prunes spoil after 2 or 3 months due to production of hydrogen swells. This does not occur in the juice prepared by whey extraction due apparently to some inhibiting substance in the whey.

Tomato beverage may be simply prepared by mixing 65 percent tomato juice, 34.6 percent whey which contains fat as whey cream and 0.4 percent salt. Fresh sweet whey should be used and it should contain sufficient whey cream to give the finished mixture a milk fat content of 2 percent to 2½ percent. Acid whey could be used if the acid fermentation is not accompanied by the production of off-flavored whey. The tomato whey mixture with a reaction of pH 4.3 to 4.5 should be heated to 140°F, homogenized at 2500 pounds pressure, canned and sterilized at 200°F for 25 minutes. Homogenization produces a smooth body but it makes the bright tomato color light, by increasing the dispersion of the suspended ingredients. This product may be used as a cold drink or as a hot tomato soup.

*Whey soups* can be prepared according to the formulas given in Table 31. When difficulties with casein coagulation are encountered in milk soups, whey substituted for all or part of the milk will often remedy the trouble.

*Whey sauce* for cream-style vegetables such as peas and mushrooms may be made from whey cream of 8 percent to 12 percent fat. From 3 percent to 5 percent starch or flour and 1 percent salt is added to the whey cream and the mixture is heated to 160°F. The sauce and vegetables are canned and sterilized according to the process required by the product being treated. Exact processing schedules are not available for different vegetables packed with a whey cream sauce but the treatment should be more severe than processes used for water packs.[127] The sauce itself can be heated to 240°F for 90 minutes without having serious damage occur to its body or storage characteristics.

TABLE 32. *Formulas for Fruit Whips*

| Flavor | Fruit Juice & Pulp, % | Acidifying Juice | | Whey Solids, % | Sugar, % | Gelatin, % |
|---|---|---|---|---|---|---|
| | | Pineapple, % | Lemon, % | | | |
| Apricot | 25 | 6.5 | 1.0 | 14 | 16 | 0.9 |
| Prune | 68 | | 1.5 | 14 | 10 | 0.5 |
| Raspberry | 30 | | | 14 | 24 | 0.9 |

Make up each formula to 100% by weight with water. Heat the mixture, with stirring, to 160°F, can, and process it at 185°F for 30 minutes. The reaction will vary between pH 4.2–4.5. To prepare the product for use, whip it vigorously for 2 to 3 minutes at room temperature, then place it in a refrigerator until served, preferably within an hour after whipping.

*Fruit Whips.* The high foaming properties of whey combined with its tolerance of a highly acid reaction suggest the possibility that whey might be substituted for egg white in fruit whips. Some formulas which were developed for such products are presented in Table 32.[176] Plain or sweetened condensed whey or a soluble dry whey are suitable sources of whey solids. Freshly made fruit whips will develop from 150 to 300 percent overrun after they are whipped with an electric mixer for 2 to 3 minutes. The first appearance of drainage from the whips will occur in from 30 to 90 minutes after whipping unless the product is cooled at once to set the gelatin. The storage properties of canned fruit whips must be improved if they are to become useful commercial products. Their whipping ability is gradually lost during a storage period of 6 to 8 months at room temperature.

*Custard type fillings* which use whey solids to supplement or replace egg white, offer possibilities in new food development. If whey solids and a pulpy fruit or vegetable are processed together, a custard like structure can be produced by the coagulation of the whey protein on the pulpy material. The accompanying formula for a prepared pumpkin pudding or pie filling is illustrative. Mix together the following ingredients by weight: cooked and mashed pumpkin 55 percent, whey solids 8 percent, refined sugar 12 percent, brown sugar 6 percent, milk fat (from whey cream) 7 percent, flavoring and spices 2 percent, water 10 percent. Heat with stirring to 185°F, can and sterilize at 240°F for 60 minutes. The mixture is of custard consistency and may be spread directly on a crust in pie preparation.

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# 9

# MISCELLANEOUS PRODUCTS

## INTRODUCTION

Milk and its byproducts are used in the manufacture of a wide variety of prepared and packaged foods. In some products such as egg or meat substitutes, milk constituents replace more expensive ingredients; but milk is generally used in foods because it improves the body and flavor characteristics of the product to which it is added. Sometimes milk byproducts are used as components of certain dietary foods prepared for persons in particular age groups.

A mixture of approximately equal weights of dried skim milk and sugar which can be quickly dispersed in water has been advocated for mass feeding of people in cases of disaster.[85] Such a mixture was successfully used for the initial feeding of Allied military personnel recovered from enemy prison and concentration camps. A combined sugar and skim milk food appears to be especially useful in the gastrointestinal disturbances that accompany radiation sickness.[85]

Concentrated forms of byproducts are needed in the preparation of most foods. The food manufacturer can choose from many kinds of bulk packed concentrates that are available in wholesale channels. The homemaker, however, whose potential requirements for milk products in home prepared foods is very great, is limited in her selection to the forms of milk she can purchase in retail stores. These include sweetened condensed and evaporated whole milk and recently, in a few markets, dried skim milk. Recipes for domestic use of dried skim milk are available.[3, 45, 54, 69] The school lunch program is utilizing large quantities of dried skim milk and there are recipes for the preparation of school luncheon dishes.[7, 8]

The observation that the fat of evaporated milk does not become oxidized, apparently because of the reducing materials developed during sterilization of the milk, has suggested to many workers that heat treated milk products might be useful antioxidants. The preparation of antioxidant materials by heat treatment of skim milk, buttermilk and particularly whey products has been the subject of several patents granted to Musher.[44] The skim milk or whey at a concentration of 25 to 30 percent solids is caramelized to produce the antioxidant material by heating it to 245°F for 30 minutes. Recently, another approach has been made to this problem. The preparation of an antioxidant for use in milk and butter, by repeated alkaline treatment of concentrated or dried whey, has been reported.[35]

## FROZEN DESSERTS

Skim milk, buttermilk and whey are used in varying degree in the manufacture of ice cream and sherbet. It is not the intent to discuss these uses in detail here but merely to mention them briefly and to indicate that such usages may now be, or may become, important outlets for these byproducts. Unusual types of mixes that may be considered as byproducts, or that utilize byproducts are discussed elsewhere: condensed mix in Chapter 4, dried mix in Chapter 5, and canned mix in Chapter 8.

**Skim Milk.** Skim milk in its different forms is a normal constituent of ice cream mix. The processes of manufacture of the skim milk products have been presented in Chapters 4 and 5. For convenience, a tabulation of normal conditions for the preparation and use of skim milk in ice cream is presented in Table 33. Some special forms of skim milk such as dispersible casein products and enzyme hydrolyzed low lactose milk are not included in Table 33, but are discussed in Chapter 4.

**Buttermilk.** Buttermilk has been utilized to a limited extent, but with considerable success, as an ice cream ingredient. It has not been widely used because only relatively small quantities of the required high grade of sweet cream buttermilk are produced. Neutralized sour cream buttermilk is of poor quality, carries objectionable flavors and is not suitable as a constituent of ice cream. Satisfactory buttermilk can be obtained from cream with a titratable acidity of less than 0.25 percent.[88]

Buttermilk is difficult to preserve without deterioration. Fresh and plain condensed buttermilk are perishable. Dried sweet cream buttermilk quickly becomes oxidized and has a shorter storage life than dried whole milk. Preliminary investigations indicate that sweetened condensed buttermilk may be a satisfactory form in which to store sweet cream buttermilk for use in ice cream or confections.[65, 88]

Buttermilk improves the whipping properties of ice cream mixes, probably because of its high lecithin content. Lecithin is associated with the milk fat and is concentrated in the buttermilk during churning. Buttermilk has been reported as improving the whipping properties of mixes.[17, 75, 86] Drum dried buttermilk,[70] fresh, plain or sweetened condensed, or spray dried buttermilk improved whipping when they replaced most of the skim milk solids generally used in the mix.[88] Others have found the improvement in whipping to be insignificant under their experimental conditions, when buttermilk was used in the mix with butter,[72] or when dried buttermilk was incorporated in ice cream mix.[13]

| Kind of Skim-milk Concentrate | Process of Manufacture | | | | | Characteristics of Finished Product | | | | Satisfactory Storage | | Relative Merits for Ice-cream use | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Forewarming Treatment | | Sugar Added per 100 pounds of Skim Milk, Pounds | Total Solids Content after Concentration, Percent | Treatment of Concentrate | Composition | | | Viscosity, Centipoises | Temperature, °F | Time, Weeks | Advantages | Disadvantages |
| | Temperature, °F | Time, Minutes | | | | Solids-not-fat, Percent | Sugar, Percent | Water, Percent | | | | | |
| Condensed | | | | | | | | | | | | | |
| Plain | 160 | 20 | 0 | 30 | Cool to 40°F | 30 | 0 | 70 | 70 | 40 | 2 | Fresh product easy to prepare | Perishable |
| Superheated | 160 | 20 | 0 | 30 | Heat to 190°F until thickened; cool by vacuum | 30 | 0 | 70 | 40,000 | 40 | 2 | Makes viscous smooth mix | Difficult to handle; viscous; perishable |
| Frozen | 145 | 30 | 0 | 30 | Cool to 34°F; freeze at −10°F | 30 | 0 | 70 | 100 | 0 | 12 | Suitable for storage | Protein gradually becomes insoluble during storage |
| Low-lactose | 145 | 10 | 5.9 | 68 | Hold quiescent at 68°F for 20 hours; centrifuge to remove crystallized lactose | 33 | 30 | 37 | 20,000 | 40 | 4 | 62% of lactose removed; can be used to raise milk-solids-not-fat of mix | Viscous; semi-perishable |
| Sweetened | 180 | 15 | 12.8 | 72 | Continuously cool to 60°F, or cool in vat with stirring to 86°F to crystallize lactose | 30 | 42 | 28 | 8,000 | 60 | 52 | Suitable for storage and retention of milk flavor | Viscosity increases in storage; may become difficult to handle |
| Dried | 160 | 20 | 0 | 40 | Heat to 160°F or higher, and spray dry | 97 | 0 | 3 | — | 70 | 52 | Especially suitable for long-term storage | Requires reconstitution; develops stale flavor in storage |

Flavor, body and texture improvements can be made in an ice cream when high quality sweet cream buttermilk is used in the mix. Buttermilk imparts a rich creamy flavor[71] to ice cream.[87, 88] Its strong emulsifying properties are instrumental in developing a smooth body and texture and in helping to retard the growth of large ice crystals during ice cream hardening.

TABLE 34. *Composition of Whey Sherbets Made from Four Sources of Whey Solids*[57]

To avoid excessive overrun, each sherbet should contain 1 percent fat added as cream. 2.5 pounds of 40 percent or 5 pounds of 20 percent cream may be used in each formula.

| Source of Whey in Sherbet | Ingredient | Weight (pounds) | Directions for Mixing |
|---|---|---|---|
| Fluid whey, 6.4% total solids | Cane sugar | 21.0 | Blend the sugars and stabilizer, and add to the fresh fluid whey. Pasteurize, and cool. |
| | Corn sugar | 7.0 | |
| | Fresh fluid whey | 71.5 | |
| | Stabilizer (pectin or gelatin) | .5 | |
| Plain condensed whey, 60% total solids | Water | 63.17 | Combine the water and whey. Blend the sugars and stabilizer and add to the water-whey mixture before it reaches pasteurizing temperature. Pasteurize, and cool. |
| | Cane sugar | 21.00 | |
| | Corn sugar | 7.00 | |
| | Plain condensed whey | 8.33 | |
| | Stabilizer (pectin or gelatin) | .50 | |
| Sweetened condensed whey, 38% cane sugar, 38% whey solids | Water | 63.35 | Combine the water and whey. Blend the sugars and stabilizer and add to the water-whey mixture before it reaches pasteurizing temperature. Pasteurize, and cool. |
| | Cane sugar | 16.00 | |
| | Corn sugar | 7.00 | |
| | Sweetened condensed whey | 13.15 | |
| | Stabilizer (pectin or gelatin) | .50 | |
| Dried whey, 95% total solids | Water (room temperature) | 66.25 | Blend the sugars, dried whey, and stabilizer. Add this mixture to the water slowly, while agitating or stirring the mixture. Pasteurize, and cool. |
| | Cane sugar | 21.00 | |
| | Corn sugar | 7.00 | |
| | Dried whey | 5.25 | |
| | Stabilizer (pectin or gelatin) | .50 | |

**Whey.** Whey, in the fresh form, or as plain or sweetened condensed or dried whey, has been used as the source of milk-derived solids in sherbet.[25, 27, 58] Whey improves the body and texture of sherbet by pro-

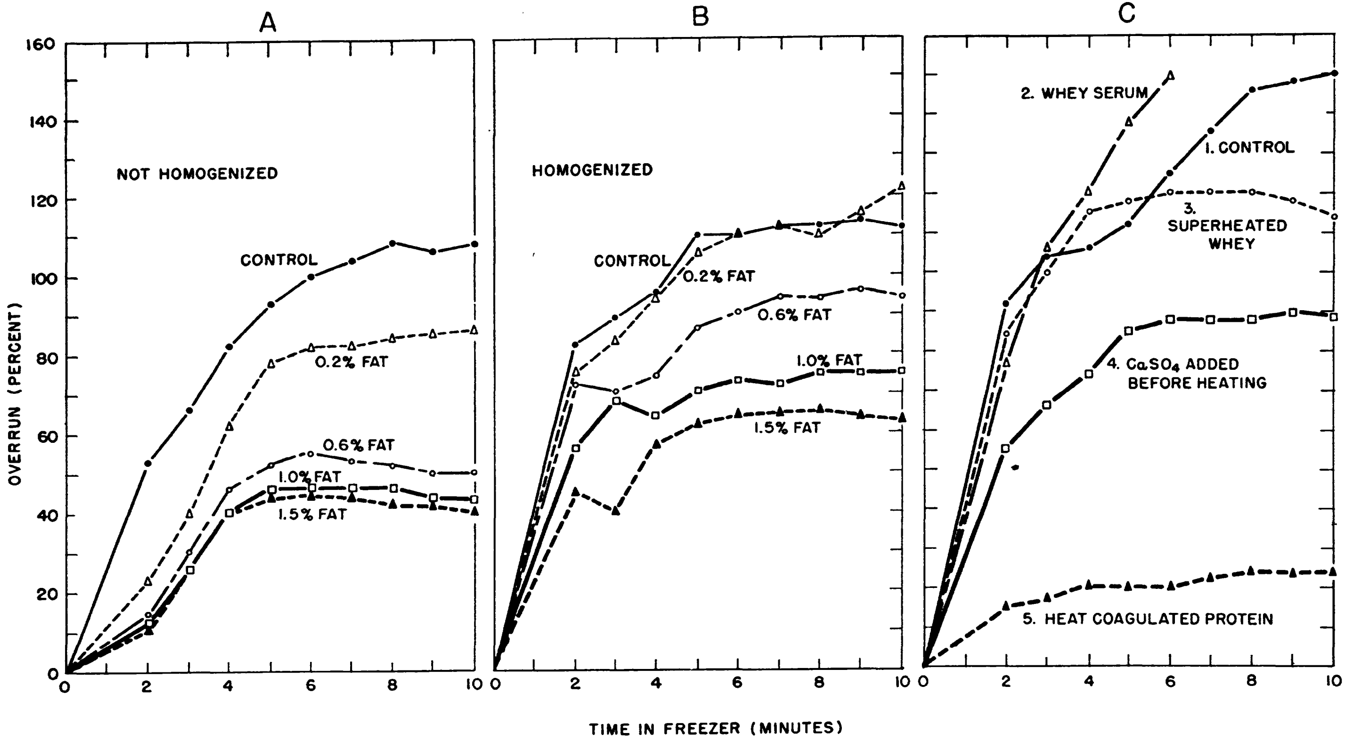

Fig. 18. A & B, The effect of homogenization of whey sherbets containing different percentages of fat upon their whipping properties. C, The effects of various treatments of whey and whey constituents upon the whipping properties of sherbets containing these products: (1) fresh cheddar cheese whey; (2) whey from which the protein had been removed after heating at 190°F for 10 minutes; (3) whey condensed to 40% total solids and heated in a vacuum pan to 200°F; (4) whey containing 330 grams calcium sulfate per 100 pounds, heated to 208°F for 10 minutes, and condensed to 30% total solids; and (5) protein removed after heating whey to 190°F for 10 minutes, dispersed in a sugar base, and homogenized.

ducing greater smoothness than is possessed by normal sherbet made with ice cream mix.

The composition of whey sherbets made from various sources of whey is shown in Table 34.[57] The sherbets contain 4 to 5 percent whey solids. When cottage cheese whey is used, the customary addition of citric acid to fruit-flavored sherbets may be greatly reduced or eliminated.

Potter and Williams[57] found that whey greatly improved the whipping properties of sherbets. Excessive incorporation of air during freezing was avoided by the addition of 0.6 to 2 percent fat to the sherbet mix. The depressing effect of fat upon the development of overrun in whey sherbet and the weakening of this effect by homogenization is shown in Figure 18, A and B. An analysis of the effect of the constituents of whey upon the whipping properties of sherbets showed that the presence of heat-coagulated whey protein decreased whipping and that the non-heat-coagulable nitrogen fraction of the serum was the active foam-producing agent. Figure 18, part C shows this relationship. The use of whey in sherbet made it possible to attain adequate overrun under composition or freezing conditions which produced a depressing effect upon the whipping of non-whey mixes.

## CONFECTIONS

The byproducts of milk used most extensively in confections are derived from skim milk and to a much lesser extent buttermilk. Recent experimental work has shown that whey also may be used but because of its low protein content it is inferior for some candies to those milk products that contain casein. Nevertheless, a good grade of candy can be made with a whey product as the dairy ingredient. Candy of highest quality contains milk fat derived from whole milk products, cream or butter.

The candy industry, perhaps more than any other segment of the food industry, has demonstrated by actual usage the great value of milk fat as a flavoring ingredient. The utilization figures given in Table 35 show that, in terms of fluid equivalents, 273 million pounds of skim milk and 211 million pounds of whole milk were used for candy in 1947. Whole milk comprised about 44 percent of the total milk utilized in confections. This is in sharp contrast to figures given in Chapter 7 which show that in terms of fluid equivalents about 4 percent of the milk used by the baking industry was whole milk.

Candy making is, to some extent, an art taught by apprenticeship and experience with less fundamental scientific background than has been accumulated by some other industries. Nevertheless, the industry has made remarkable progress in the production of stable, uniform and attractive goods. The milk products used by confectioners are those that are commercially available to the food industry. The dairy and the candy industries have made little effort to determine what kind of treatment should go into the manufacture of milk products that are to be used in candy. The heat and other treatments incidental to preparation of the concentrated and dried milks can greatly affect the physical characteristics of finished dairy products and their behavior in some of the candy making processes. It seems reasonable to assume that if the two industries determined the basic physical and chemical requirements of good dairy ingredients and if such ingredients were produced especially for candymakers, greater utilization of milk products in candy would result. Speical products made in the past for candy use have often been from secret formulas, unavailable to the average candymaker.

TABLE 35. *Production of Milk and Some Milk Products* and Their Utilization in the Confectionery Industry*

| Milk Product | Production 1947 | | Confectionery Industry | |
|---|---|---|---|---|
| | Product, Million lbs. | Fluid Equivalent, Million lbs. | Product, Million lbs. | Fluid Equivalent, Million lbs. |
| Plain condensed whole | 175 | 367 | 5.567 | 11.681 |
| Plain condensed skim | 402 | 1,206 | 12.438 | 37.314 |
| Sweetened condensed whole | 247 | 544 | 40.699 | 89.538 |
| Sweetened condensed skim | 543 | 1,629 | 38.110 | 114.330 |
| Dried whole | 165 | 1,319 | 13.211 | 105.688 |
| Dried skim | 678 | 7,701 | 11.908 | 121.462 |
| Fluid whole | 119,300 | 119,300 | 3.972 | 3.972 |
| Form not classified | | | 15.613 | |

* Exclusive of cream and butter.

Data based on estimates of production, U. S. Bureau of Agricultural Economics; and estimates of confectionery utilization, U. S. Department of Commerce.

Milks products usually appear in candy formulas in the condensed or dried form. It is more efficient for the confectioner to pay the milk processor to concentrate milk in large scale equipment than it is to have his candymaker boil off the water in small batches. Furthermore, excessive boiling of thin, dilute mixtures that contain milk protein may cause the protein to coagulate in large aggregates, thus producing a coarse texture in the candy. The manufacture of the standard milk concentrates is described in other chapters. Confectioners sometimes order special concentrates to meet the requirements of individual formulas. These usually have not deviated greatly from conventional milk products. One such "plastic" or high-solids sweetened condensed milk contains approximately 13 percent milk fat, 35–37 percent milk-solids-not-fat, 30 percent sugar and 20–22 percent water.[65] Richmond[60] has discussed the use of milk products in fudge and caramel.

**Functions of Milk Constituents in Candy.** Each milk constituent, except the minerals, can be shown to perform useful functions in candy other than the recognized addition to nutritive value. The lactose, protein and fat are also responsible for certain types of deterioration. There is no evidence to show that the minerals of milk play a significant role in the physicochemical problems of the candymaker. They may, however, affect the behavior of the protein during the cooking process, and they also contribute a certain characteristic saltiness which helps produce a well rounded milk flavor.

*Lactose* is the least sweet of the sugars that are used in confections. In a scale of sweetness in which sucrose is 100, lactose is 16, glucose 70, levulose 173 and corn sirup 30.[10] Lactose can thus aid in decreasing the sweetness of oversweet candy.

Lactose is less soluble than the other common sugars. In the supersaturated state it will crystallize readily if free moisture is available to satisfy the requirement of a molecule of water of crystallization per molecule of lactose. If the sirup phase of fondant is supersaturated with respect to lactose this will often cause a sandy texture to develop during storage. However, Paine and associates[53] were able to produce an attractive, mildly sweet, cream fondant by crystallizing pure lactose in an invert sugar sirup.

Large quantities of lactose may be used to advantage in fudge if provision is made to crystallize the lactose during graining of the fudge. The use of excessive quantities of lactose in caramels may induce crystallization, and before the lactose begins to crystallize, the caramel can be very sticky. This sticky characteristic will disappear with age as crystallization sets in. Stickiness will be avoided if the lactose is added to the candy as a component of milk or skim milk rather than whey. The relatively large portion of protein associated with the lactose in the milk products helps to inhibit lactose crystallization. Whey, being low in protein and high in lactose, will produce a sticky caramel that will grain easily unless the whey solids content of the caramel is held below 15 percent or unless additional milk solids are used in the formula.

The physical state of lactose in candy may have an important effect on the apparent freshness of some confections. As long as the lactose remains amorphous there will be softness, elasticity and freshness of body. If crystallization proceeds slowly during storage, the structure may become crumbly, dry and hard. A gradual crystallization of lactose in candy can be avoided by so choosing the ingredients that there is an ample supply of inhibiting materials such as invert and corn sirup, protein and fat. Some of these substances should be present even in whey fudge. To obtain fine granular structure in whey fudge the lactose should be quickly crystallized so as to produce small crystals.

Lactose is known to adsorb riboflavin and other pigments on its crystal faces during crystallization. This adsorptive power may enable the crystallized lactose of fudge type candies to hold vanilla and various flavors and pigments longer than when the candy contains no lactose.

Refined lactose is too expensive for fondant or special usage while technical grades that contain appreciable quantities of protein often foam excessively during cooking. This foaming can be eliminated by digesting the whey protein with trypsin prior to separation of the lactose.[83]

The presence of milk protein and lactose together in a white fondant eventually causes discoloration in storage. However, the association of protein and lactose during cooking helps to produce the attractive brown color of many candies.

*Milk protein.* The confectioner depends upon protein to produce foam in the manufacture of marshmallow, nougat creams and mazettas. Gelatin and egg albumin are the favorite proteins for this purpose although whey protein can be used. Most whey protein preparations are rather salty in flavor and inferior in foam production to gelatin and egg white. However, attractive whey candies that depend upon whey protein for air incorporation will be described later in this chapter.

Milk protein is essential to the development of proper body, flavor and color in such cooked candies as caramel and fudge. The favorable characteristics produced by the milk protein are largely developed in the candy mass during the cooking process.

Casein and whey protein function differently in candy. Casein is capable of forming a firm, resilient, chewy type of body which is neither sticky nor tough. As the candy mass boils in the cooking process, it is essential that the casein coagulate in a finely dispersed state to insure smoothness. Coagulation appears inevitable because the casein is destabilized both by high cooking temperatures and by high concentrations of proteins, sugars, salts, and other ingredients.

The fineness of the casein coagulum is governed by the conditions present at the time of coagulation, such as the speed of agitation just preceding and during coagulation, the viscosity of the hot mass and the concentration of casein in the mixture. It has been shown[81, 82] that severe agitation during coagulation of concentrated milks produces a finely dispersed coagulum but one of lower viscosity than when the heating is done with little or no agitation. The viscosity of the boiling candy mixture is usually great enough to insure the formation of a fine protein coagulum provided its formation is accompanied by vigorous stirring. The viscosity of the mixture may be increased by increasing its protein content or by adding such body building ingredients as dextrin, starch or other cereal products.

Buttermilk protein is less effective in building firmness of body in cooked candy than skim milk protein because it contains a different protein fraction from that of skim milk. Buttermilk produces the same firming effect on the body of candy as a mixture of skim milk and whey. Buttermilk, because of its high lecithin content, is more useful than skim milk in helping to form smooth emulsions and in producing a creamy body.

Whey protein forms a soft flocculent coagulum which is less capable of contributing firmness to the structure of candy than casein. Indeed, 40 percent of the nitrogen in whey is present in soluble, non-heat-coagulable form and this material does not aid in the development of a firm structure.

The whey proteins surpass casein in foaming characteristics. At high concentrations and viscosities, such as may be found in sweetened condensed whey, a moderately stiff foam of good stability can be formed. A whipped whey or milk albumin foam cannot be set by heat, a property unique with egg white.[12] A coagulable whey-protein foaming agent can be produced from a condensed whey paste.[39] Part of the lactose is removed from the condensed whey, the whey is diluted with water, made alkaline, treated with a flocculating agent, acidified and filtered. The clear liquor contains the active foam-producing constituents of the whey and imparts foaming properties to aqueous solutions.

Whey foam can be used to introduce air in a candy mixture but means must be provided to stabilize the structure and prevent escape of the air. This can be accomplished by first whipping the whey product and then carefully folding it into the cooked and partially cooled candy or sirup base. The foam will be quite stable if the base contains no fat. The hardening mixture entraps and permanently holds the air. This type of treatment is well illustrated by the process for making "whipped fudge" described in the candy formula section. Since fat quickly destabilizes a whey foam, any fatty ingredients such as chocolate should be carefully folded into the mixture at the latest possible moment before it becomes too cool and viscous for proper mixing.

A porous confection can be made from whipped sweetened condensed whey by drying the foam before it can collapse. The whipped mixture may be stabilized by the addition of a precooked cereal. However, the product will become sticky and finally the lactose will crystallize if it is allowed to take up moisture. Chocolate coating is effective in retarding moisture absorption.

The color of highly cooked candies that contain milk is caused by the familiar browning reaction between protein and the sugars of the mixture. The pleasing caramel flavor develops simultaneously with the color. The intensity of this milk-caramel flavor is partly dependent upon the quantity of milk protein used in the candy.

*Milk fat* cannot be omitted from a discussion of candy ingredients although it is often absent or present only in limited amounts in byproducts. Milk fat is a relatively soft fat with a melting point between 82 and 97°F. A blend of milk fat and of a confectioner's higher melting point fat is often used to give firmness to the candy. Milk fat itself performs the usual functions of any fat of the same melting point, that of building up a pliable, plastic body.

The most important function of milk fat in candy is to transfer milk flavor from milk to candy. Candy flavor is improved more by the addition of milk fat to the candy base than by the use of any other ingredient. Milk fat can also cause serious flavor deterioration in candy. Rancidity due to hydrolysis and tallowiness caused by oxidation are more sharply defined in milk fat than in most other types of fat. These defects should not occur in candy, at least during several months of storage, if a good quality milk fat is used and if the candy ingredients are well cooked during manufacture. Rancidity is usually caused by enzyme action or high acidity in the milk product. When milk fat is allowed to absorb oxygen, oxidation and a tallowy flavor develop. If high quality milk fat is used, the candy maker can rely on the heat treatment used in cooking the candy to destroy enzymes and to produce reducing materials which will protect the milk fat during the normal life of the candy.

Lecithin is associated with the fat fraction of milk, but when cream is churned, lecithin is concentrated in the buttermilk. Lecithin is an emulsifier and occurs to the extent of 1.6 percent in dried buttermilk. The emulsifying property of buttermilk makes it especially useful in the manufacture of such confections as milk chocolate and butterscotch.

**Milk and Whey Candy Formulas.** Representative formulas are presented in Tables 35 and 36 to demonstrate the use of milk byproducts in the more common types of candy. Many variations can be made in such formulas. Confectioners often use secret formulas which produce candies suitable for their own special markets. Two groups of formulas are given. Those in Table 35 require skim or whole milk products while those in Table 36 require whey.

**Procedure in Using Milk Candy Formulas** (Table 36). The caramel and fudge should be stirred during cooking to prevent burning. Water is given in some of the formulas to aid in preparing a homogeneous mixture prior to boiling. When a dried milk product is used, water is generally necessary to disperse it. The plain condensed and evaporated milks contain 64 to 74 percent water. They may be substituted in the formulas for the sweetened condensed or dried milks, but if this is done the other materials should be combined with only about one-third of the fluid milk product and the mixture started to boil. The remaining milk should be added gradually to the viscous, boiling batch. If this is not carefully done, the milk may coagulate as large objectionable aggregates of curd. The curd particles are difficult to disperse and they produce an unfavorable effect on the appearance, taste and texture of the candy. For best results the milk protein must be so treated that it coagulates as very small aggregates. These minute particles of curd form during rapid stirring in a thick or viscous mixture. They are at once protected by a coating of the other

TABLE 36. *Milk Candy Formulas*

A tabulation of the quantities of chief ingredients needed to make some representative candies. For working directions for each candy refer to the text. Add ½ to 1 oz. of vanilla to each batch to suit the taste.

| Ingredients | Weight of Ingredients | | | | | | |
|---|---|---|---|---|---|---|---|
| | Pan Caramel, lbs. | Milk Caramel, lbs. | Caramel Toffee, lbs. | Fudge A, lbs. | Fudge B, lbs. | Handroll Creams, oz. | Marshmallow, oz. |
| Sugar (brown for coffee) | 32 | | 34 | 31 | 25 | 36 | 56¾ |
| Corn sirup | 32 | 31 | 34 | 21½ | 2½ | | 24 |
| Invert sirup | | | | 3 | | 5 | 24 |
| Hardened vegetable fat | 10 | 4 | 12 | 1¼ | | | |
| Dried skim milk | 15 | | | | 2 | 5 | 11½ |
| Sweetened condensed whole milk | | 65 | 20 | 12 | | | |
| Water | (21) | | | (5) | (10) | (7½) | (38½) |
| Special ingredients | stabilizer | emulsifier | | | | invertase, citric acid | gelatin, pectin, invertase |
| Fondant | | | | 21½ | 2½ | | |
| Frappé | 11 | | | 9¼ | | 4 | |
| Salt | | | | ½ | | ⅛ | ½ |
| Weight of ingredients except water | 100 | 100 | 100 | 100 | 32 | 50⅛ | 115¾ |
| Source from which formulas were adapted: | 5 | 63 | 4 | 9 | 19 | 27 | 40 |

candy ingredients and this prevents them from combining with other curd particles to produce a noticeable coagulum. When a high concentration of milk solids from sweetened condensed or dried milk is used, a viscous mixture is obtained at once. Long boiling to remove excess water is avoided and a spontaneous type of coagulation occurs which produces a soft, flocculent and easily dispersed precipitate.

Varying quantities of fat may be used in the formulas. If a richer candy is desired the fat should be increased. The milk fat may be 12 to 16 percent for caramels and 16 to 18 percent for toffee.[66] However, rancidity in high-fat candies is still a problem. The fat to milk-solids-not-fat ratio of the formulas of Table 35 should preferably remain unchanged when one milk product is substituted for another. A vegetable fat may be substituted for milk fat but only by sacrificing quality and flavor.

Either condensed or dried forms of buttermilk and skim milk may be used in the formulas if adjustments are made for the sugar and milk solids content of these products. Blending of ingredients and cooking processes must be conducted in a manner to suit the form of milk byproduct used in the formula. Buttermilk solids contain about 1.6 percent lecithin, which is useful as an emulsifier in such candies as caramel, butterscotch, or milk chocolate.

Whey should not be substituted for milk in the milk candy formulas. The high lactose and low protein content of whey usually requires that special whey candy formulas be used when whey is to be one of the chief candy ingredients.

*Pan caramel* (Table 36).[5] Mix the sugar and dried skim milk, then add one ounce of stabilizer (CaO, $Na_2CO_3$, or MgO). Gradually add the water to form a paste, then a semi-liquid. Heat slowly, then boil and add the fat. Add the corn sirup after the mixture reaches 225°F. Mix in the frappé when the batch reaches the desired firmness.

*Toffee* and milk caramels (Table 36)[4, 63] are made by cooking the ingredients to about 245°F, or to the firmness desired. A trace of lecithin or 0.5 percent sorbitol monostearate may be used as an emulsifier in the milk caramels.

*Milk fudge A* (Table 36).[9] Cook all ingrdeients together except the fondant, salt and frappé. After the boiling mixture reaches 238–240°F, cool 3 or 4 minutes and add fondant, salt and frappé. Cast in starch.

*Milk fudge B* (Table 36).[19] Mix the sugar and skim milk solids, gradually add water, first to a paste, then to a semi-liquid mass. Heat slowly to boiling, then add corn sirup and cook to 230 to 235°F. Cool to 170°F, add fondant, mix, add salt and vanilla to taste, pour on oiled paper.

*Handroll creams* (Table 36).[27] Thoroughly mix the dried milk and sugar; then add the water and invert sugar. Heat slowly in a steam jacketed kettle to melt the sugar grain, then boil to a soft ball. While creaming, add salt, vanilla, 3 grams of invertase, 1½ grams of 50-percent citric acid solution and the frappé. Finish creaming, handroll, and chocolate dip. Six ounces of dried buttermilk may be substituted for the 5 ounces of dried skim milk. Only 3 ounces of frappé is needed for the buttermilk creams.

*Marshmallow* (Table 36).[40] Dissolve 1¼ ounces granulated gelatin in 1 pound cool water, let set 30 minutes. Heat the following: 1½ pound corn sirup and 1½ pound invert sugar to 190°F, add 1 pound fine sugar to melt. Cool to 150°F, add gelatin solution. Beat for 20 minutes, slack back with 2½ pounds sugar sirup, then add to batch the non-fat milk solution, pectin solution, 4 grams invertase, ½ ounce salt, and 1 ounce vanilla. Marshmallow to be cast with oval top in dry starch. Let set till next day, remove from starch and chocolate dip.

Sugar sirup: 2½ pounds sugar, 1¼ pounds water, heat to 190°F, cool to 125°F.

Milk solution: 11½ ounces non-fat dried milk, 1¼ pound sugar sirup.

Pectin solution: Mix dry ¾ ounce sugar, 3 grams pectin, add to 2½ ounces hot water.

**Procedure in Using Whey Candy Formulas** (Table 37).[76] The formulas of Table 37 are calculated for the use of sweetened condensed whey as the source of whey solids. Plain condensed or dried whey may be substituted for the sweetened condensed product which contains 38 to 40 percent whey solids and an equal quantity of sugar. When sweetened condensed whey is not used, the quantity of sugar normally included in it may be mixed with the plain condensed or dried whey before these are added to the candy batch. Since sugar aids in the wetting of dried whey, sugar-dried-whey mixtures should be prepared and dispersed in water at room temperature. The whey-sugar solution may then be added to the candy mass.

The fudge, caramel, and taffy (Table 37) should be cooked in a kettle having a fast double-action stirrer. Water may be added before cooking if necessary to insure proper mixing. The coagulum formed by whey protein is soft and flocculent. It requires no special treatment during cooking and disperses well in the candy mixture. When whey is the only source of milk protein there is no tendency toward the development of curd lumps or of an irregular rough texture as is sometimes caused by casein coagulation when skim milk is used.

*"Wheyfers"* (Table 37). Whip the sweetened condensed whey in a mechanical beater for 5 minutes or more until its volume is at least doubled. The addition of materials containing fat or oil must be avoided, since they destabilize the whip. Spread the nuts on fine-mesh-screen drying-trays.

Carefully fold the cereal into the whipped whey and extrude the whip on the screens in ribbons 1½ inches wide and ¼ inch thick. Dry the mixture in a tunnel drier at 210°F for about 1 hour. The drying time will depend upon the temperature, the air circulation, and the thickness of the candy. Score, cool, and coat with chocolate to prevent moisture absorption.

*Whipped whey fudge* (Table 37). Cook the sugar, corn sirup and invert sirup to 272°F, allow to cool to 230°F. Whip the sweetened condensed whey in a mechanical beater for at least 5 minutes. Mix the cooled sirup, whipped whey, and vanilla, and carefully fold in the melted chocolate. Avoid excessive stirring of the finished mixture. Pour in wooden forms.

TABLE 37. *Whey Candy Formulas*[76, 80]

| Ingredients | "Wheyfer" Ingredients, lbs. | Whipped Fudge Ingredients, lbs. | Fudge Ingredients, lbs. | Caramel Ingredients, lbs. | Taffy Ingredients, lbs. |
|---|---|---|---|---|---|
| Sweetened condensed whey | 84 | 32 | 43.0 | 45 | 52 |
| Sugar | | 40 | 11.0 | | |
| Corn sirup | | 16 | 9.0 | 28 | 42 |
| Invert sirup | | 5 | 3.0 | 6 | |
| Skim milk solids | | | | 6 | |
| Hardened vegetable fat | | | | 4 | |
| Milk fat (as butter or cream) | | | 2.5 | 5 | |
| Precooked dry cereal | 4 | | | | |
| Fondant | | | 20.0 | | |
| Chocolate | | 7 | 6.0 | | 6 |
| Powdered lactose (for seed) | | | .1 | | |
| Nuts (optional) | 12 | | 5.4 | 6 | |
| Vanilla to flavor | | | | | |
| Total weight of ingredients | 100 | 100 | 100.0 | 100 | 100 |

The percentages of total candy solids derived from the whey are:
For "wheyfers," 40%; whipped fudge, 14%; fudge, 20%; caramel, 21%; taffy, 26%

*Whey fudge* (Table 37). Cook (with stirring) the sweetened condensed whey, sugar, half the corn sirup, and the invert sirup. The cream or butter may be added after the sirup has been partly boiled down. Cook to 248°F. Cool 25 or 30 degrees or transfer to smaller pouring kettles, add the remaining corn sirup, fondant, and chocolate, and stir well for several minutes. Add the powdered lactose, flavoring, and nuts, and stir for several minutes. Pour into wooden forms.

*Whey caramel* (Table 37). Cook the ingredients with stirring to about 246°F. The milk products may be added after the sirup has been partly boiled down. Add nuts and flavoring before pouring on a stone slab.

*Whey taffy* (Table 37). Cook the sweetened condensed whey and sugar to 248°F, or higher, if desired; cool, add chocolate, and pull.

**Miscellaneous Confections.** The products and byproducts of milk are used in the manufacture of a wide variety of confections. Only a few will be mentioned in this miscellaneous grouping to illustrate some common as well as some unusual candy products. Much could probably be done to develop new and attractive confectionery mixtures that would be useful to the candy manufacturer or that might appeal to the consumer.

*Chocolate products* contain milk and milk byproducts in accordance with the definitions and standards promulgated by the Food and Drug Administration.[20] Detailed information concerning the kinds of milk and other ingredients used in chocolate products may be obtained by consulting a copy of the regulations.

Milk chocolate must contain not less then 3.66 percent by weight of milk fat and not less than 12 percent by weight of milk solids. The weight of the non-fat milk solids must be not more than 2.43 times and not less than 1.20 times the weight of milk fat. This in effect requires that milk chocolate be made from a whole milk product or its equivalent in terms of fat and solids. Dried or sweetened condensed whole milk are generally used for chocolate manufacture. The condensed milk product should be low in moisture, hence specially prepared sweetened condensed milks of 75 to 80 percent solids or even higher are sometimes made.

Skim milk chocolate contains less than 3.66 percent of milk fat and not less than 12 percent of skim milk solids. Buttermilk chocolate contains buttermilk solids in place of skim milk solids. Mixed dairy product chocolate is made from various dairy ingredients and contains less than 3.66 percent of milk fat and not less than 12 percent of milk constituent solids.

Dried sweet cream buttermilk has been suggested as an emulsifying ingredient in the manufacture of chocolate coatings.[64]

An important phase of milk chocolate manufacture is the mixing and blending of ingredients. The milk products and chocolate are intimately blended by a conching or mixing operation that results in a coalescence of particles and the formation of a smooth homogeneous structure. A full development of milk flavor occurs in the conching operation. Sweetened condensed milk products are well suited to chocolate manufacture since they merge with the other ingredients to yield a smooth mass of proper viscosity and pronounced milk flavor. When dried milk is used it is sometimes necessary to add extra cocoa butter in order to secure the same homogeneous blend in the product as is obtained with the sweetened condensed milks. The fat in a mixture of spray-dried skim milk and spray-dried cream has the property, when warm, of coalescence on trituration and the dried-skim-cream mixture is therefore better for chocolate manufacture than dried whole milk.[46]

*Chewing gum* is made from a blend of several kinds of chicle flavored to suit the taste. Sweetened condensed milk is often used in chewing gum in quantities up to about 5 percent of the weight of the gum. The condensed milk improves flavor and aids in body development. Chicle itself is rubbery but the addition of sweetened condensed milk makes it softer and more pliable and prevents the gum from breaking down excessively in the mouth.

*Fountain sirups* for topping ice cream are made with various forms of milk products. A hot fudge topping may be prepared by first mixing 4 pounds of sugar, 14 ounces of cocoa and a gallon of milk, or 12 ounces of dried skim milk plus 3 quarts of water. Boil the mixture 10–15 minutes until smooth. Dissolve 1 ounce of cornstarch in a pint of cold water and stir slowly into the chocolate mixture. Add 3 ounces of butter (5 ounces if dried skim milk was used) and heat with stirring until desired thickness is reached. Strain if mix is not smooth. Add vanilla to taste.

Caramel topping may be made[5] by mixing 10 pounds of sugar, $3\frac{3}{4}$ pounds of dried skim milk, $5\frac{1}{2}$ pounds of water and 10 grams of a stabilizer ($CaO$, $Na_2CO_3$ or $MgO$) to form a smooth semi-liquid mass. Heat to boiling with stirring and add 10 pounds of corn sirup, continuing to boil the batch to 218–220°F. Salt, vanilla and butter may be added to taste.

An excellent sirup or topping may be made by mixing a chocolate, caramel, coffee or other flavored non-acid base with a sweetened condensed milk product. Sweetened condensed whole, skim or buttermilk or sweetened condensed whey may be used. The quality of the topping will depend in large measure upon the quality of the sweetened condensed product. The flavor base can be made with little or no milk but it should contain a sugar in water concentration of 60 percent. Mixtures of various quantities of flavor base and sweetened condensed product may be prepared to suit flavor and viscosity requirements. It is not necessary to cook the topping since each component contains enough sugar to inhibit bacterial growth. Since the sweetened condensed product is not cooked it is capable of transferring a full milk flavor to the topping. A viscous but stable sweetened condensed product in which the lactose crystals are small and uniform in size is required.

*Milk-honey confection*, a candy developed by Iverson,[30] is made by drum drying a mixture of 100 pounds of whole milk and 9.4 pounds of honey. The milk is pasteurized at 170°F, homogenized, cooled to 60°F and mixed with the honey. The mixture is dried on drums 28 inches in diameter revolving at 12 revolutions per minute and maintained at 270°F. The plastic mass is scraped from the drums, formed into bars and chocolate coated. The low moisture content of the product and the presence of the invert sugar in the honey are effective factors in keeping the lactose in an amorphous, non-crystalline condition.

*Peanut butter-whey mix.* A mixture of equal parts of peanut butter and sweetened condensed whey will produce an attractive sweet filling or candy center.[79] It may also be used as a sweet spread. The addition of sweetened condensed whey takes the sticky, clinging characteristic from peanut butter and gives it a smooth, short type of body. The mixture is prepared by blending one pound of sweetened condensed whey for each pound of peanut butter to be treated.

*Fruit mixtures.* Sweetened condensed whey may also be mixed with various fruit jams for use in spreads or toppings. The physical qualities of such mixtures are satisfactory since the fruit acid will not coagulate the whey protein. However, as is often the case with whey mixtures, the fruit flavor is diluted by the whey and is inferior to the flavor of the original fruit.

An apple-milk confection that will utilize surplus and cull apples has been prepared by mixing pulped apples and skim milk and drying the mixture on a vacuum drum drier.[74]

*Dried honey-skim milk mixture* is a powder that may be pressed to yield an attractive honey-flavored wafer or that may be used as a source of honey and milk solids in candy manufacture or for the preparation of other foods, including dry mixes and bakery goods. This product was developed to fill the need of food manufacturers for a dried honey.[84] The mixture is prepared by either of two methods. Equal parts of honey and concentrated skim milk (35 percent solids) on a solids basis are spray-dried by conventional methods. This mixture is hygroscopic and difficult to handle.

A second and better process for preparing a dried honey-milk mixture is to pour, drip, or spray honey slowly into dried skim milk with constant, slow agitation. A product containing 50 to 60 parts of honey solids to 40 to 50 parts of skim milk solids can readily be made. The honey droplets immediately become surrounded by a coating of dried skim milk. Spray dried milk in which the lactose is amorphous must be used. After the honey has been stirred into the milk powder, the mix should be seeded with lactose and dextrose and spread on trays. Both the lactose from the milk and the dextrose from the honey crystallize with absorption of one molecule of water obtained from the moisture of the honey. If the mixing has been properly done a light powder will result.

Crystallization may continue for an hour or more after which the product may be dried in the trays to insure good keeping quality. Although the lactose and dextrose of the mixture are crystallized, the levulose of the honey remains amorphous and capable of some water absorption.

**Indian Confections.** Milk and certain indigenous dairy products are used extensively in the manufacture of the special confections found throughout India. Ghee, or Indian milk fat, is prepared by heating country butter to temperatures as high as 240°F and separating the fat. Ghee is used as a component of many native candies. Several milk preparations of a concentrated nature that might properly be considered as byproducts are also used either in confections or without further processing, as sweets themselves. Detailed information on native Indian products may be obtained from Davies' pamphlet.[18] Sen[62] furnished the author some of the material included in this discussion.

The fluid milk of India is often produced and handled under unsanitary conditions. To safeguard this milk against spoilage and to protect the health of the people, bulk milk is always boiled before it is used. This habit of boiling the milk no doubt led to the development of the native milk products, most of which are prepared on a domestic scale by drastic heat treatments.

The preparation of Indian confections is a secret art which is practiced by individual specialists. The candies may contain some form of milk product, cereal and sugar. The ingredients are cooked as small batches in open kettles. Individual characteristics in the confections are developed by variations in type and quantities of materials, cooking conditions and physical manipulations. It is hoped that a brief description of a few typical Indian milk products may initiate thinking along new lines that might help to evolve a new approach to byproduct utilization in American confections. The composition of some concentrated Indian milk products used for confections is given in Table 38. Actually there are wide variations in composition and methods of preparation but the values of Table 38 may be considered as representative of average products.

*Channa* is the product resulting from the acid precipitation of the protein from heated milk. The fat is removed with the protein coagulum while the whey is discarded. A large amount of Indian raw milk is used for channa. The finished confection is made by adding sugar to the casein-fat mixture.

One method of making channa is to pour whey of 0.80 percent titratable acidity onto boiling milk. The curd is gathered, drained, pressed and macerated. The resulting, damp, finely divided curd or channa is quite perishable. Channa is preserved by making it into a variety of sweets, all of which contain sugar. For example, rasa geolla is made by grinding channa and semolina together, forming the mass into balls and boiling these in a sugar sirup.

*Malai*, sometimes called sar, is the clotted cream layer that forms on the surface of heated milk. Whole milk is simmered all day and the layer of cream and coagulated protein, sometimes one inch thick, is removed as malai. Milk of high fat content, such as Indian buffalo milk and milk with large fat globules, gives the best and largest yields of malai. According to Davies[18] the approximate composition of the milk skin on a dry basis will be: fat—55 percent, protein—36 percent, and mineral matter, mostly calcium phosphate—9 percent. If sugar is added during the heating process a thicker layer of malai can be recovered. Unsweetened malai resembles clotted cream and is used on sweets and puddings. Layers of the sweetened variety are rolled up and used as confections.

*Rabbri* is prepared by skimming boiling sweetened milk in somewhat the same way that malai is made. The skins are removed continuously and collected on the cooler parts of the pan. Twenty-five to 40 minutes of slow simmering are required to collect the creamy skins as they form.

TABLE 38. *Approximate Composition of Indian Milk Products Used as Confections or in the Manufacture of Confections*[1]

| Product | Treatment while Heating | Protein, % | Fat, % | Lactose, % | Ash, % | Sucrose, % | Water, % |
|---|---|---|---|---|---|---|---|
| Channa | acid precipitation | 18.5 | 32.5 | 2.3 | 0.3 | — | 46.5 |
| Malai | surface skimming | 3.2 | 28 | 3.4 | 0.4 | — | 65 |
| Rabbri | surface skimming | 9.5 | 19 | 16.0 | 3.0 | 25 | 28 |
| Kheer | evaporation | 9.0 | 17 | 14.0 | 2.4 | 20 | 38 |
| Khoa | evaporation | 18.0 | 36 | 30.0 | 5.0 | — | 11 |
| Khoa[2] | evaporation | 24.5 | 27 | 20.0 | 4.0 | — | 24.5 |

[1] This table was compiled largely from data given by Davies.[18]

[2] Suggested minimum standard.[31]

*Keer* is a form of sweetened condensed milk but it is a perishable product since its sugar-in-water content is less than 40 percent. Keer is made by concentrating sweetened milk in open kettles.

*Khoa* is the native dried milk of Central India. It may vary in moisture content from 10 to 30 percent. Khoa is used as a constituent of sweets when it may be cooked in sirup or it may form a soft filling in sweet goods. Small batches of milk and sugar are boiled together with constant stirring in shallow round-bottomed iron pans. Close attention is required in the final stages of heating. When the product begins to dry in the pan, it is gathered together in a doughlike mass and formed into pats for market.

## MEAT PRODUCTS

Concentrated forms of milk, usually dried skim milk, may be used in the manufacture of special meat products and meat substitutes. Meat products designed for interstate shipment must conform with the requirements of the meat inspection service of the Bureau of Animal Industry.[11]

The total cereal, starches and non-fat milk solids must not exceed 3½ percent of the weight of finished sausage. Meat loaves are made with either pork or beef and the moisture limit is 3 percent. This imposes an automatic limit on the quantity of milk solids that can be used. Excessive amounts of skim milk will produce a dry crumbly loaf. Usually each pound of non-fat milk solids will absorb one pound of water. A third class of meat product may be labeled "Loaf" followed by a statement of the optional ingredients it contains. A "Loaf" may contain any quantity of skim milk solids but if more than 12 percent is used the meat flavor will be very weak. Meat substitutes must be properly labeled with a statement of ingredients as required by the Federal Food, Drug and Cosmetic Act.

Skim milk solids compare favorably in nutritive value with the types of meat used in sausage and meat loaves. Dried skim milk contains about 36 percent protein while the grades of beef, pork and veal used for sausage contain approximately 53, 13, and 69 percent protein, respectively. There probably is little difference in the relative nutritive values of the meat and milk proteins.

The presence of skim milk solids apparently has a tendency to aid in the development of an "off" or stale flavor during the storage of bulk or loaf sausage but this does not occur in those meat products that are filled into casings and processed.

The chief physical function of skim milk in chopped meat products is that of taking up water and at the same time contributing to the production of a firm texture. During cooking in the presence of the other sausage constituents the casein is denatured with an apparent absorption of water. The resulting finely disperesed curd blends well with the chopped meat and helps to bind it together to produce a smooth but chewy meat-like texture. This reduces crumbling, improves slicing properties and cuts down shrinkage during storage. Certain cereal products may be capable of greater water absorption but the water absorbing properties of skim milk are ample for good quality sausage. From considerations of the effect on nutritive value, flavor, texture, and water binding capacity, skim milk solids can be considered better for use in sausage and meat loaves than cereal products.

*Preparation of meat products.* Little information is available on the actual processes used in the preparation of sausage, meat loaves, and related products. The primary object in manufacturing these foods is to make a nutritious and attractive food from what is usually regarded as an unpalatable part of the animal carcass. The processing generally consists of mincing or grinding the meat, mixing the water absorbing agent (cereal or milk byproduct) and seasoning with it, molding the mixture or filling it into casings and cooking the product.[59]

Flavoring with spices, herbs and various seasoning materials is a very important part of sausage manufacture. In some parts of England, for example, strong sage and thyme flavors are preferred while in other parts cayenne pepper or cinnamon are favored.[59] Continental sausages are made with a variety of flavorings such as peppers, nutmeg, mace, cinnamon and thyme. They are processed to produce certain delicate and characteristic flavors.

The non-meat ingredients may consist of one or more cereals such as wheat rusk, wheat flour, farina, barley, oatmeal, or rice. Milk byproducts are highly favored members of the non-meat, water absorbing group of ingredients.

Sausages may be cooked in open tanks of water but care must be taken to control the process so as to avoid bursting of the sausage casings. The canned meats are cooked in the usual type of pressure autoclave.

Smoked and cooked sausage such as bologna, salami, and frankfurter are treated according to the results desired. The meat mixture is cured with sodium nitrate and sugar, stuffed into casings and smoked. Cooking is then done by heating in hot water until the interior of the sausage reaches 160–180°F.

*Formulas.* The following formulas taken from a group of 65 published by the American Dry Milk Institute[52] will serve to illustrate the use of milk solids in meat products:

### Frankfurter[52]

| | | | |
|---|---|---|---|
| 45 | lbs. veal trimmings | 2 | oz. mace |
| 20 | lbs. bull meat | 4 | oz. ground mustard |
| 35 | lbs. pork trimmings | 1 | oz. onion powder |
| 4½ | lbs. non-fat milk solids | 1¼ | oz. garlic |
| 3 | lbs. salt | 2 | oz. paprika |
| 1 | qt. cure | 36 | lbs water |
| 7 | oz. white pepper | | sheep casings |

The cure consists of a mixture of 3 lbs. 7 oz. sodium nitrate, 5 oz. sodium nitrite and 10 lbs. of dextrose made up to 5 gal. with water. The batch is smoked 1¾ hrs. at 130–175°F, cooked and cooled. The loss in weight during processing is 9 percent.

### Liverwurst[52]

| | | | | |
|---|---|---|---|---|
| 40 | lbs. hog livers (scalded) | | 4 | lbs. onions (fresh peeled) |
| 15 | lbs. hog gut fat (scalded) | | 3 | lbs. salt |
| 15 | lbs. hog tripe | cooked until tender | 6 | oz. white pepper |
| 20 | lbs. hog skins | | 1½ | oz. sweet marjoram |
| 10 | lbs. beef tripe | | ½ | oz. ground cloves |
| 4 | lbs. non-fat milk solids | | | |

Cook skins and tripe slowly in water and remove; scald livers and fat in same broth. Cut skins, add liver, tripe and onions and chop, then add fat, non-fat milk solids and spices, chop until fine, mix with broth and stuff in beef rounds. Cook 30 minutes, chill and hang to dry.

Roast Beef Loaf[52]

100 lbs. fresh beef
3 oz. Worcestershire sauce
10 lbs. tomato catsup
3 lbs. salt
8 oz. white pepper
2 oz. ground bay leaves
2 lbs. grated onions
12 lbs. non-fat milk solids

Cut meat in ½-inch pieces, cover with water in steam jacketed kettle, bring to boil, add all ingredients except skim milk, cook until tender. Place meat in mixer with non-fat milk solids and add 45–50 lbs. of cooking broth, mix, place in pans and chill.

## MEAT SUBSTITUTES

Many attempts have been made to utilize milk protein in the preparation of meat substitutes. These trials appear to have met with little success, judging from the scarcity of published results, or of commercial production of actual products. Two characteristics, the flavor and texture of meat, have not yet been successfully produced by processing milk protein. There is little difference, however, in the nutritive value of the meat and milk proteins.

The appealing odor and flavor of cooked meat cannot be developed by any known treatment of milk protein. Sodium glutamate, a product of casein hydrolysis, has been said to have a meat-like odor. However, its addition to a variety of animal and vegetable products has shown that rather than contributing a flavor of its own, sodium glutamate tends to intensify the natural flavor of the food to which it is added. The products of casein hydrolysis can be successfully used in gravies or meat sauces but they do not contribute meat flavor in the absence of meat.

The texture of meat has not been duplicated in any kind of milk protein preparation. However, it is reasonable to suppose that processes could be developed which might produce in casein the chewy, structural properties that are characteristic of chopped meats or hamburger. An approach to this kind of texture is made in the preparation of cottage cheese. If the cottage cheese granules were firmed by extra cooking and extrusion of whey, a toughened, granular casein could be produced which might be suitable for incorporation in a chopped meat-like product.

Dried skim milk can be successfully added to meat-type loaves in rather large quantities but meat should be present to give flavor. Cooking will cause the milk protein to coagulate, absorb water and help to produce a firm texture. Such products must be properly named and labeled.

Sure[68] has developed a processed, meatless dairy food that contains principally a blend of dried milk solids, dried yeast and whole wheat or polished rice. The mixture results in a grayish powder to which an equal weight of water is added just before it is cooked. Some of the ingredients of the finished loaf are 21.9 percent protein, 2.2 percent fat, 4.9 percent ash, 0.64 percent calcium, 0.027 percent iron, 1.77 percent fiber, 54.6 percent water and an abundance of vitamins.

A meat substitute has been reported to have been prepared from skim milk in Germany.[26] Skim milk protein is fortified with dried blood and fat to improve texture, color, flavor and caloric value. The mixture is heated, extruded under pressure and the threads are compressed tightly into loaf form and cellophane-wrapped. The fat acts as a lubricant and extrusion produces a fibrous or grain-like texture.

## EGG SUBSTITUTES

Attempts have been made to produce egg substitutes from milk protein but the results have not been encouraging. Preparations with good whipping properties that contain casein generally have an alkaline reaction. The whey protein fraction possesses excellent foaming characteristics that can be used where the foam is not to be set by heat. But whey protein lacks the ability to form, by whipping and heat coagulation, a fine stable foam structure, a characteristic which makes egg white a unique food product.

Peter and Bell[56] studied the whipping properties of whey protein solutions prepared from the concentrated whey liquor that is a byproduct of lactose manufacture. The whipping properties of the liquor were increased when small amounts of calcium hydroxide were added to the whey liquor before it was heated to 131°F for 10 minutes. The alkali was neutralized and the product spray dried to yield a whipping compound for food use.

Clickner[14] extracted the lipoid material from whey powder, then added the powder to egg white as a substitute for the yolk. The whey-egg white mixture was said to equal whole egg in emulsifying properties.

A variety of egg substitutes were developed and used during World War II. One of these was made by neutralizing a skim milk and whey mixture with a calcium salt and then concentrating it.[15] Wheat or soybean flour to the extent of 30 percent of the total solids of the mixture was sometimes used as a thickener.[61]

Another product that could be whipped to a stable foam was prepared by concentrating 1,000 kg. of skim milk to 38 percent solids, then stirring 0.176 kg. of rennet extract into the concentrate. After a gel structure formed, a mixture of 0.88 kg. of acid sodium pyrophosphate and 0.354 kg. of sodium hydroxide dissolved in 8 liters of water was added. The mass was held 4 hours at 95°F, cooled to 65°F and the reaction adjusted to pH

6.5 with dilute sodium hydroxide. The product was then homogenized and roller or spray dried.[36]

Egg substitutes were made in Germany during World War II from skim milk and whey.[6, 55] Two types have been described. Milei G is an egg-yolk or whole-egg substitute which is said to have the binding properties of egg in bakery products and household cooking. Milei W is an egg-white substitute which has the whipping properties of egg-white and is used mainly for meringue-type bakery goods.

Milei G is produced by first adjusting the titratable acidity of skim milk to 0.1575 percent (7° Soxhlet-Henkel) with sodium bicarbonate, and then condensing it under vacuum to 40 percent solids. A low concentration at this stage gives a fluffy finished product preferred for baking purposes, while a high concentration produces a dense powder. The concentrated skim milk is cooled to 55°F and run into a mixing kettle with a propeller-type agitator where the following ingredients are added: (a) a slurry of bean meal (6 kg. per 1,000 l. of milk), (b) water-soluble yellow color, (c) disodium phosphate to bring the mix to pH 7.5. The product is roller dried to 3 to 4 percent moisture, sieved through a coarse screen and packed in small paper containers.

Milei W is made either with skim milk or a mixture of 2 parts of skim milk with 1 part of whey neutralized with sodium hydroxide. The byproduct is condensed under vacuum to 40 percent solids, the reaction adjusted to pH 8 to 9 with milk of lime, and the mixture is then spray dried to 3 to 4 percent moisture. Milei W is more hygroscopic than Milei G and cannot be roller dried because of the sticky film which is formed when the pH is high.

## MARGARINE

Margarine is composed largely of vegetable oils and some animal fats emulsified with skim milk, milk or cream. It may, or may not, also contain a special emulsifier and some starter distillate with other flavoring ingredients. Milk fat is rarely used in margarine but all manufacturers rely upon emulsifying their oils with non-fat milk solids. Skim milk is used in the fluid or dried form. Dried skim milk has the advantage of being easily shipped, stored and handled and it may be made up to a higher solids content than fresh skim milk. The skim milk to be used in margarine is pasteurized and cultured with a lactic starter to develop about 0.7 percent titratable acidity. Approximately 80 percent vegetable oil, 18 percent skim milk and 2 percent salt are used. The milk-solids-not-fat of the finished product may vary from 1.8 to 3.0 percent. The emulsion is prepared at 110°F, then chilled to 35°F to form the margarine.

Four general methods of margarine manufacture are in use:[73] (a) The wet method. The oil and skim milk are proportioned into a mixing conveyer, then dropped into a tank of water held at 35° to 40°F. The quick chilling crystallizes the fat into granules after which the margarine is tempered, printed and packaged.

(b) Refrigerated drum method. The oil and skim milk are premixed and dropped onto refrigerated double drums similar to the type of drum used to dry milk. The thin sheet of emulsified oil hardens on the drums as it cools to 35°F during one revolution of the rolls. The margarine is scraped from the drums, tempered and formed into prints at 40°F.

(c) Continuous chilling method. The oil-skim milk mixture is emulsified and continuously chilled by forcing it through a machine similar to the continuous ice cream freezer. The margarine emerges from the chiller ready to print.

(d) Churning. The oils and skim milk are emulsified to produce a cream-like product which is cooled and churned just as butter is churned. When the emulsion breaks the margarine is worked and printed.

## DRESSINGS FOR SALADS

A recently promulgated definition and standard of identity for mayonnaise, French dressing, and salad dressing excludes milk products from the list of permissible ingredients that may be used in the manufacture of these foods.[21] However, milk products have been included among the ingredients of special dressings for salads. Care must be exercised to label such a dressing in a way that will not represent it as one of the defined products mentioned above.

Egg-yolk is the only emulsifying agent used in mayonnaise. Nor have the emulsifying properties of egg yolk ever been equaled by any milk product. Clickner[14] used whey to improve the emulsifying power of egg white. He prepared a lipoid-free dried whey by extracting whey powder with a fat solvent such as isopropyl alcohol. When 10 pounds of this finely pulverized whey powder was added to 80 pounds of whole egg material, the mixture was said to be equal in emulsifying power to egg yolk.

Soluble whey protein was advocated for use as a whipping and emulsifying agent in dressings for salads.[56] The product was prepared from the concentrated whey protein solutions that were the mother liquors left from a process of manufacturing lactose which did not denature the protein. The whipping properties of the liquor were improved by the addition of small amounts of calcium hydroxide followed by heating at 132°F for 10 minutes and neutralization.

Whey or skim milk have been used as a substrate for mold growth after which the wet mold is dispersed by heating to give a white acid emulsion which is said to resemble mayonnaise in taste and consistency.[55] Milk has been advocated as an important ingredient in dressings for salads[16] and highly heat treated milk, a non-allergic product for persons who are allergic to egg yolk, has been mentioned as an emulsifier to replace the yolk.[22]

Dried skim milk may be used as an ingredient for dressings for salads.[3] When skim milk of 25 to 30 percent solids content is whipped for a few minutes a stiff white foam is formed.[77] The foam can be set by the addition of acid from vinegar or fruit juice, but subsequent disturbance of this whip will cause wheying off. Foams set with a stabilizer have greater stability.

## ION EXCHANGE PRODUCTS

The Zeolite process of softening hard water is a relatively old one but the application of the ionic exchange principle to milk products was first made by Lyman and co-workers in 1933.[37, 38] At that time the milk products were contacted with various base-exchange silicates but in recent years carbonaceous and synthetic resinous exchangers have been developed. Lyman's early work was chiefly concerned with the removal of both calcium and phosphate ions from milk to produce a soft curd product. New exchange materials can now be used to alter greatly the ionic equilibria of milk and whey products. The field is being actively explored and the new techniques will aid in fundamental studies of the ionic changes that occur in the processing and storage of dairy products. Commercial applications, while still limited, can be expected to follow the research work.

Garrett has presented a simple explanation of the ion exchange process and has shown its application to dairy processing.[24] Many papers on various phases of ion exchange have appeared in the chemical journals, and the annual unit process reviews appearing in the January issues of Industrial and Engineering Chemistry include reviews of ion exchange work.

There are two general types of ion exchangers. The cationic exchanger exchanges its own positive elements, such as sodium, calcium, magnesium or hydrogen, for cations in the solution which is passed over it. The anionic exchanger exchanges its negative radicals, such as chloride, sulfate, phosphate, citrate or hydroxyl ions for anions in solution. An amine type of exchanger apparently combines chemically with most acid molecules. Water may be purified by first passing it through a bed of cationic exchanger material that has been regenerated to act in the hydrogen cycle. The mineral cations in the water such as calcium and sodium are replaced by hydrogen ions which produce an acid reaction. The water is passed over an anionic exchanger which removes the sulfuric, hydrochloric or other acids that have formed from the anions present in the water.

Similarly, ionic exchangers are used to alter the ionic equilibria of fluid milk products, principally skim milk and whey.

**Ion Exchange Procedures.** It was necessary to solve many problems before the ion exchange procedure could be successfully applied to milk and whey. However, processes for treating these products and for regererating the exchangers are now available. Treatment may be by either of two methods. The batch process consists in mixing the milk and exchange material, then separating them by filtration. The column process involves passage of the milk through a column or bed of the exchanger.

*Use of synthetic inorganic exchangers.* During some of the early work natural greensand was found to be unsatisfactory for the treatment of milk.[48] However, a hydrated synthetic sodium aluminum silicate known as Crystalite showed a high capacity of calcium removal, uniformity and purity. Otting[48] points out that many difficulties arose even with the use of this material. As a result of much research and refinement of methods it is now possible to use Crystalite daily for more than a year with but little replacement. It will treat 125 gallons of milk per cubic foot of exchange material. Milk can be produced which is uniform in color, flavor, acidity and curd tension, at less than 0.5 cents per quart. Approximately 20 percent of the calcium and phosphorus present in the milk is removed by the sodium aluminum silicate.[48]

Milk, to be treated for removal of calcium and phosphorus, is carefully acidified at 50°F with citric acid to produce a titratable acidity of 0.25 to 0.35 percent but without the development of a coagulum. It is then passed through the exchanger and the removal of calcium and phosphorus is accompanied by a reduction to normal acidity.[29]

The following procedure, described by Otting,[48] may be used to revive the spent exchange material:

(a) The Crystalite is washed upflow with 100°F water to remove any residual milk.

(b) A wetting agent (sodium lauryl sulfate, 0.005 to 0.1 percent) is circulated upflow for 10 minutes. This removes fat, protein and some phosphorus.

(c) Rinse upflow with water.

(d) Pass downflow a 5 percent solution of sodium chloride acidified with acetic acid and buffered with sodium acetate to pH 4.4.

(e) Rinse downflow with water.

(f) Circulate a sodium hydroxide-sodium aluminate solution downflow.

(g) Rinse downflow with water and leave Crystalite covered with water which will be forced out by milk at the beginning of the next run.

*Use of organic resin exchangers.* Haller[28] has investigated both the use and regeneration of synthetic organic resins when applied to the alteration of the ionic equilibrium of milk and whey.

For calcium, or calcium and phosphate, removal a cation exchanger of the sulfonic acid type is used. The amount of calcium that can be removed is dependent upon the exchange capacity of the resin. The methods used for regeneration of the resin determine what ions will be removed and the resulting effect on the pH of the milk. The reaction of the milk must be returned to its pre-treatment value by chemical means or by a compensating ion-exchange treatment.

Four general types of treatment have been outlined by Haller, the first of which is an original method:[28]

(a) Regenerate the resin with 4 percent sodium chloride solution and adjust it to pH 2.25 with hydrochloric acid. Passage of the milk over this resin will remove up to 50 percent of its calcium and close control or adjustment of the pH of the milk is not necessary. However, by varying the pH of the regenerant, Haller has been able to remove different amounts of phosphate. In such cases the resulting low pH of the milk should be readjusted to a normal value by one of the methods used in c or d below.

(b) Treat the milk with a resin regenerated in the sodium cycle by 4 percent sodium chloride solution. The alkalinity of the milk is then carefully reduced by batch treatment with a cation exchanger until its reaction returns to normal. The second exchanger is one that has been regenerated in the hydrogen cycle with 4 percent hydrochloric acid. This method removes both calcium and phosphate.

(c) The milk is first carefully acidified by citric or sulfuric acid or it is treated with a cation exchanger regenerated in the hydrogen cycle (4 percent HCl) until its titratable acidity reaches 0.35 percent or less, depending upon the amount of calcium to be removed. If acidification of the milk is performed by a resin, with its simultaneous removal of sodium, the sodium content of the final product will be lower than if an acid acidulant is used. The acidified milk is then brought to a normal reaction by treatment with an exchanger regenerated in the sodium cycle. Both calcium and phosphate are removed.

(d) The milk is acidified by an exchange resin as in method c. The pH of the milk is restored to normal by treating it with an amine-formaldehyde type of anion exchanger regenerated by 4 percent sodium carbonate solution. Both calcium and phosphate are removed.

**Ion Exchange in the Processing of Milk Products.** The ionic equilibrium of milk products can be adjusted by standardization with ion-exchange-treated skim milk or whey. The addition of suitable amounts of one of these treated byproducts to a mixture during processing can strikingly affect certain properties of the finished product. The treatment of whole milk involves certain mechanical and technical difficulties that result in the loss of milk fat in the filter beds. The ion-exchange treatment of whole milk can usually be avoided by standardization with treated skim milk.

*Uses for ion-exchange-treated skim milk.* The first application of the base exchange treatment of milk and skim milk was in the production of a soft curd and infant milk following the work of Lyman.[37] Skim milk was treated with 20 percent or more of its weight with the wet base exchange silicate, glauconite. The CaO and $P_2O_5$ values were reduced by about 30 percent and the acidity of the milk was then brought back to normal with hydrochloric acid. The curd tension of the treated skim milk was greatly reduced and the standardization of special milks with the treated skim milk effectively produced a soft curd milk. More recently, as mentioned in a previous section, the milk has been acidified before ion exchange treatment rather than afterward.

The development of "sandiness" due to crystallization of lactose in high milk solids ice cream has been retarded by the use in the mix of a skim milk of low calcium phosphate content.[51] The skim milk is percolated through a bed of ion-exchange material to remove about 30 percent of the calcium and 20 percent of the phosphate of the milk. The treated milk is then used in the ice cream mix in fluid form or it may first be concentrated or dried. Ice creams that contained as much as 16 percent base-exchange-treated milk-solids-not-fat did not develop sandiness during storage. In explanation of this it was stated that the ion-exchange treatment caused some kind of change in the colloidal milk constituents that retarded or prevented lactose crystallization.[48] The mechanism by which partial removal of calcium phosphate from a mix can retard lactose crystallization in the ice cream is not well understood.

Evaporated milk can be stabilized toward heat by adding ion-exchange skim milk to it before sterilization at temperatures up to 270°F.[33, 49, 50] The skim milk may be prepared by passing it through a resinous cation exchanger to remove 60 percent of the calcium. Since this increases the reaction of the milk to about pH 8.0 it should be adjusted back to pH 6.6 by means of a hydrogen exchanger. The skim milk then may be used in fluid, concentrated or dried form. The low-calcium skim milk may be added to the batch of evaporated milk at any stage prior to canning of the evaporated product. Such an addition may be used in place of the customary phosphate salts to stabilize evaporated milk during sterilization. Evaporated milks requiring 2 to 7.3 ounces of disodium phosphate per 1,000 pounds of concentrate to produce proper heat stability can be similarly stabilized by treating 0.5 to 2.5 percent of the original milk by the ion exchange process or by standardizing the batch with this quantity of base-exchange concentrate. The ion-exchange method may be used to stabilize high-solids milk in the manufacture of evaporated milk of 32 to 34 percent solids. The alteration in the calcium and ash content of evaporated milk as a result of the ion-exchange treatment of a small part of the milk is insignificant.

Cream may be stabilized by ion-exchange treatment of the skim milk with which it is subsequently standardized.[23] The skim milk is subjected to cationic-exchange material acting in the alkali-metal cycle. This removes 60 to 70 percent of the calcium and gives the milk an alkaline reaction which must be corrected by passage through a hydrogen exchanger. The skim milk is then ready for standardizing cream. Through its use the calcium-phosphorus ratio should be reduced from the 1.2 of normal milk to between 0.5 to 0.75. Such stabilized cream may be standardized and dried to produce a cream with a fat content within the range of 50 to 70 percent. The fluid or dried stabilized cream will not feather, coagulate or oil off in hot coffee. It will not curdle when it is added to acid fruits, when it is used in creamed soups, or when it is boiled in caramel manufacture.

Cream standardized with as little as 5 percent of its weight of ion-exchange-treated skim milk before it was forewarmed and homogenized reduced its feathering tendency and increased its stability to sterilization by heat.[34]

Atmospheric drum-dried skim milk is highly insoluble and therefore is priced at about 2 cents per pound below the soluble spray-dried product. Stevens and Haller,[67] in a preliminary investigation, found that removal of part of the calcium of skim milk by ion exchange made it possible to dry some milks on an atmospheric drum without greatly impairing their solubilities. If work in progress confirms this finding with all skim milks, it may be possible to produce drum-dried milk that compares favorably in quality to the spray product.

*Ion-exchange-treated whey products.* A new approach to the recovery of substantially all the lactose in whey has been made by Almy and Garrett.[2] Skim milk or whey is treated with exchange material in the hydrogen cycle to reduce the reaction to pH 4.6–4.8. The protein is coagulated by heating to 175–210°F and then removed. The whey is again contacted with exchange resin in the hydrogen cycle to reduce the reaction to pH 1.6–1.8. This treatment removes 90–95 percent of the metal ions. The demineralized whey is next passed through an anion-removing exchanger to raise the reaction to pH 7.5 to 9.0. After adjustment of the reaction to about pH 6.6 the liquor is concentrated and dried to 1.4 percent moisture.

The finished product contains 95.2 percent lactose, 2.6 percent non-coagulable protein and 0.8 percent ash.

It seems possible that ion-exchange treatment of whey might be useful in the conventional processes of lactose manufacture. Recent investigations into the use of ion-exchange treatment of sugar liquors showed that 70 to 80 percent of the nitrogen compounds of the sugar beet juice were removable by a cation bed and 20 to 30 percent by an anion bed.[32] Results of somewhat the same order for the reduction of the non-protein nitrogen in deproteinized whey have been reported by Miller and Arrigoni.[43]

Synthetic resins remove color from sugar liquors, anion exchangers being more effective than cation exchangers in this respect.[1]

A demineralized, deacidified whey, useful in food manufacture, has been prepared by Meade and Clary.[41] Whey is subjected to repeated cycles involving successive contacts with a decationizing medium and a deacidifying medium. The flow over the different resins is so regulated that the reaction of the whey during treatment remains within the limits pH 3.8 to 7.5. The modified whey may be concentrated or dried after treatment. A typical analysis of the dried product is: moisture 1.6 percent, acid 0.2 percent, lactose 84.5 percent, protein 11.2 percent, ash 3.3 percent. This modified whey is slow to develop a brown discoloration during heating or storage apparently because of its reduced amino acid content. The dried product is said to be relatively non-hygroscopic, and when heat treated or held in storage it will not develop a cooked or stale flavor. It is especially useful for the preparation of infant or special milks and milk concentrates, to which it imparts heat and storage stability.[42]

*Casein and soluble caseinates* have been improved by means of ion exchange treatment.[47] A suspension of commercial casein is treated with a base exchange material to produce a soluble sodium caseinate of very low calcium and phosphorus content. The sodium caseinate may in turn be precipitated by acid to yield a casein which does not show a determinable amount of calcium. These unusually pure, low ash products are advocated for special food and commercial uses.

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# 10

# CASEIN AND WHEY PROTEIN

## CASEIN

The average annual consumption of casein in the United States since 1935 has been approximately 60 million pounds. The annual domestic production during this period has varied between an oversupply of 67 million pounds from 624 plants in 1937 to the low of 12 million pounds from 47 plants in 1945. Importations from Argentina have supplemented the domestic production to the extent necessary to meet demands for consumption. Table 39 gives the production of dried casein in the eight principal producing states for the nine most recent years available, showing that more than 90 percent of domestic casein is produced in these eight states.

Diversion of domestic milk to the greatly increased production of dried milk had the effect of decreasing the 1943 domestic production of dried casein to less than half that of the immediately preceding years. The production continued to decrease to the end of the war and then began to increase somewhat. Readjustment of domestic casein production in the future will depend chiefly on the magnitude of the need for concentrated milks and on the degree of success with which other industrial proteins compete with casein for the domestic market.

Statistics show that usually nearly half of the casein produced annually in this country is made in the months of May, June, and July, that is, during the so-called "flush season," when the production of milk is at its peak. Although the larger plants operate throughout the year, in the early part of summer much more skim milk is available at dairy plants than can be utilized for perishable products, such as cottage cheese, chocolate milk, and cultured buttermilk, and hence their production of casein is increased. Furthermore, there are many dairy plants that produce casein only from the surplus skim milk of the summer months.

The quantity of casein produced in any period is influenced by the market prices of other products that can be made from milk, and by the market for casein. Whenever the prices of cheese, condensed skim milk, or dried skim milk are relatively high, some plants make little or no casein. A price for casein greater than about 15 cents per pound in prewar years stimulated its production, but a price lower than 10 cents per pound discouraged production.

About 70 percent of our casein supply is used in paper-coating, with con-

TABLE 39. *Dried Casein Produced in the Eight Principal Producing States—1940 to 1948*[1]

| | 1940 pounds | 1941 pounds | 1942 pounds | 1943 pounds | 1944 pounds | 1945 pounds | 1946 pounds | 1947 pounds | 1948 pounds |
|---|---|---|---|---|---|---|---|---|---|
| Wisconsin | 11,954,000 | 11,688,000 | 11,937,000 | 3,681,000 | 1,711,000 | 1,148,000 | 3,238,000 | 8,151,000 | 1,862,000 |
| California | 11,815,000 | 10,501,000 | 7,451,000 | 3,455,000 | 3,606,000 | 2,668,000 | 5,042,000 | 7,553,000 | 5,071,000 |
| New York | 8,076,000 | 8,665,000 | 6,568,000 | 4,041,000 | 3,944,000 | 3,668,000 | 3,359,000 | 5,369,000 | 3,126,000 |
| Minnesota | 4,824,000 | 6,287,000 | 5,925,000 | 1,773,000 | 1,224,000 | 1,374,000 | 3,351,000 | 7,650,000 | 1,714,000 |
| Idaho | 2,427,000 | 2,598,000 | 3,708,000 | 2,397,000 | 701,000 | 774,000 | 566,000 | 1,100,000 | [2] |
| Vermont | 2,380,000 | 2,018,000 | 1,981,000 | 1,424,000 | 1,371,000 | 1,312,000 | 779,000 | 889,000 | 419,000 |
| Illinois | 1,544,000 | 1,926,000 | 1,912,000 | 173,000 | 221,000 | 427,000 | 939,000 | 2,574,000 | 271,000 |
| Washington | 1,325,000 | 1,555,000 | 1,406,000 | 867,000 | 630,000 | 208,000 | 486,000 | 1,192,000 | 974,000 |
| Total for eight states | 44,345,000 | 45,238,000 | 40,888,000 | 17,811,000 | 13,408,000 | 11,579,000 | 17,760,000 | 34,478,000 | 13,437,000 |
| Total for the United States | 46,616,000 | 47,346,000 | 42,268,000 | 18,386,000 | 15,264,000 | 12,333,000 | 18,319,000 | 35,831,000 | 13,372,000 |

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[1] From reports of Bureau of Agricultural Economics, U. S. Dept. of Agriculture.

[2] Production in Idaho not separately reported and not included in state total for 1948.

sumption in casein glue, and casein paints of much less magnitude. Not only the quantities of these products being made, but also the substitution of other proteins and starch for casein, are factors in the demand for casein. Starch is used in coating paper, and soybean protein and zein are being advocated as substitutes for casein in practically all fields, but so far none of these has qualified as a substitute adequate in all respects. Current research may overcome these inadequacies, however.

All of the industrial casein produced in the United States is acid-precipitated casein, with the exception of relatively small quantities of rennet-precipitated casein produced for use in the manufacture of plastics. The acid used is either sulfuric or hydrochloric acid or lactic acid produced by fermentation of the lactose in the skim milk, or in whey that is added to the skim milk. The desired physical characteristics of the curd are obtained by regulation of the degree of acidity, the rate of stirring, and the temperature. The degree of purity desired is obtained by correlation of degree of acidity, temperature, and amount of washing.

Casein can be produced economically on a small scale as a side-line in a dairy plant that is already equipped with the necessary vats, since little additional equipment is absolutely essential and cost of labor and other charges can be kept small. It is often advisable to arrange equipment and methods of operation in a dairy-products plant so that change can be made quickly from the manufacture of one product to another without appreciably affecting efficiency of operation. It should be noted in this connection that dry casein can be stored for months without deterioration and therefore can be made in isolated areas and in seasons of flush milk supply and under other conditions that would not permit the production of perishable food products. Wet curd is perishable and hence drying equipment in or near a plant making casein is highly desirable.

If casein is the principal, or only, product to be made in a plant, more expensive and efficient equipment, such as some or all of the items of a continuous process, may be installed. It will be necessary to process at least 25,000 pounds of skim milk daily in order that costs per unit of casein produced may be kept to a minimum, and a larger supply is desirable.

The plant should be located as near the sources of milk supply as possible so that skim milk will be in sweet condition when received for processing. For large-scale operation, a separate building is desirable, but if casein manufacture is conducted as one part of a dairy enterprise, as is nearly always the case, this building should be near other units for economical transfer of skim milk, power, steam, and water. Adequate and suitable space for storage of dry casein should be included. If a separate building for casein manufacture and storage is not feasible, the space allotted to the casein operation should be separated from other departments, in so far as possible. Ample facilities for disposal for whey and wash water must be provided to avoid pollution of streams.

**Equipment for Batch Processes.**[31] The equipment for making casein by a batch process is simple and inexpensive. Some of the items are those used in making other dairy products, some can be readily constructed at the plant from materials available locally, and the few items that must be purchased can be obtained from dealers in casein or dairy equipment.

The following list includes the equipment necessary for producing dry casein by processing 10,000 pounds of skim milk per day:

- One 1,000-gallon tank of metal or wood for receiving and storing skim milk
- Two 5,000-pound, or one 10,000-pound, metal milk or cheese vats or wooden vats to hold the skim milk for precipitating, draining, and washing the curd
- One 1,000-gallon tank of metal or wood for storing whey, if lactic casein is to be made
- Simple titration equipment, or bromocresol green paper, for measuring acidity
- Two 2-inch centrifugal pumps for handling skim milk and whey
- One or two curd presses, with dividing boards and press cloths
- One curd mill of the shredder-beater type
- One hundred eighty drying trays, 30 inches square, with bottoms of 8- or 10-mesh galvanized-iron screening
- Six trucks for moving trays
- One drying tunnel, 22 feet long, 6 feet high, and 32 inches wide
- One high-speed electrically-driven fan, 30 to 35 inches in diameter, for circulating heated air
- Radiators, or steam coils, for drier, with steam trap for condensate
- One recording thermometer for use in controlling temperature of air in drier
- One grinder for dried casein
- Small items, such as strainers, steam piping, valves, steam and water hose, rakes or paddles, thermometers, buckets, bags, and scales for weighing dried casein

If hydrochloric or sulfuric casein is to be made, glass-lined or hard-rubber containers of several gallons capacity will be needed for diluting the acid, but a tank for storing whey will not be required. If the casein is to be sold unground, a grinder will not be needed; it if is to be sold as wet curd, the drying equipment also can be omitted.

*Vats.* Steam-jacketed metal cheese vats are desirable, but wooden vats provided with means of introducing live steam are much less costly and nearly as satisfactory. Single-walled iron vats can be used, but it is difficult to keep them from rusting. Redwood, cypress, yellow pine, and Douglas fir are the woods preferred for construction of vats and other wooden equipment exposed to skim milk or moist casein. Rectangular vats of approximately 5000 pounds skim milk capacity are satisfactory in plants handling small quantities of milk. If larger batches are to be processed, a better casein can be produced in less time and with less labor if the bottoms of the vats are curved and mechanical agitation is provided. Wooden vats should be made of 2-inch stock and should be braced with rods or long bolts under the bottoms and along the sides. To facilitate drainage, the bottoms of the vats should slope toward the outlets, which should be provided with gate valves. All vats should be so located that they can be easily cleaned, both inside and outside.

*Presses.* The curd press is a device for expelling mechanically from the wet curd as much moisture as possible, which is that in excess of a little more than the weight of the moisture-free casein. It is much less expensive to press out water than to evaporate it by means of artificial heat. In the United States the heat of the sun cannot be depended on for drying curd, as it is in Argentina.

Curd presses should be constructed of heavy stock and firmly bolted together, as shown in Figures 19 and 20, which illustrate the hand-operated, batch type and the 2-roll, mechanically-driven continuous type. The batch press may be purchased, or assembled at the plant. All wooden parts should be given several coats of linseed oil or other wood-sealing composition to prevent as much as possible the absorption of moisture. Even so protected, they should be scrubbed daily to prevent the development of offensive odors.

The dividing boards that are placed between the wrapped cakes of wet curd are constructed of hard wood strips ½-inch thick and 1½ inches wide and are in the form of lattices with openings one-inch square. Galvanized nails should be used and the boards treated with several coats of a sealing composition. Daily scrubbing of the boards is necessary.

Press cloths for wrapping the wet curd are commonly of burlap sacking with the edges hemmed. This material, however, is difficult to wash satisfactorily and to keep clean. Ten-ounce duck, lightweight canvas, and cloths woven and sold especially for the purpose are more suitable than burlap and will give better service.

A mechanically-driven, continuous press may be used in connection with batch precipitation and is preferable in processes involving continuous precipitation. It offers the advantages that the curd can be dried promptly after precipitating and washing, thus avoiding deterioration of the casein during long periods of pressing, and that the amount of hand labor required is decreased. The upper roll is solid and is forced against the lower roll by spring pressure. The lower roll may be either solid or perforated. Since one passage between a pair of rolls, properly adjusted as to pressure exerted, reduces the moisture percentage to approximately 55 percent and subsequent passages have slight effect on the water content of the curd, a 2-roll press is adequate. A continuous press will deliver 1,000 to 2,000 pounds of pressed curd per hour, which is equivalent to approximately half that weight of dried casein.

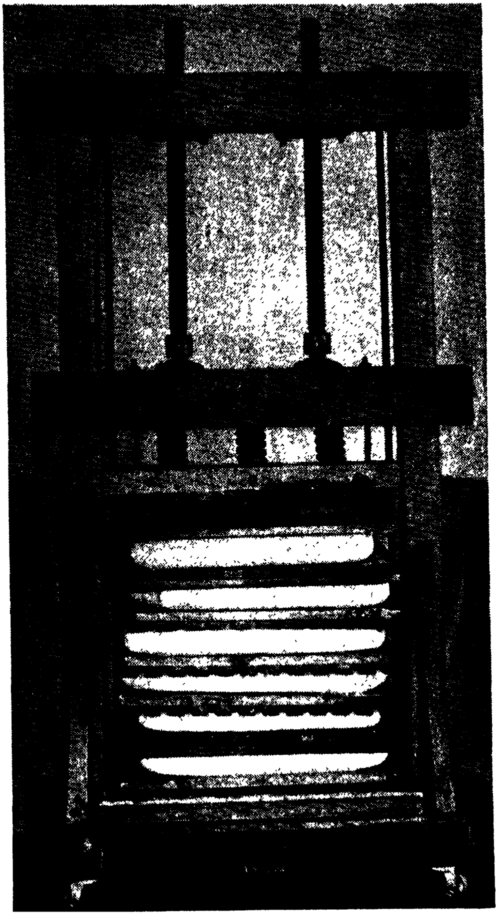

Fig. 19. Casein curd press—batch type.

Firm curds produced by some precipitation methods may be drained sufficiently for drying without the use of a press. Shallow, movable vats with bottoms of either perforated sheet iron or No. 4 galvanized screening may be used for draining, or the draining surface may be installed as a raised false bottom in a deep vat.

*Curd Mills.* The usual type of curd mill consists essentially of a shredding roll and a beating roll. The shredding roll is a metal cylinder rotating at a moderate speed and fitted with rugged, pointed pegs that cut and tear the curd into fine pieces. Below the shredding roll is the rapidly rotating beater roll which forces the shredded curd to pass through perforations in a metal drum and fall on drier trays placed beneath.

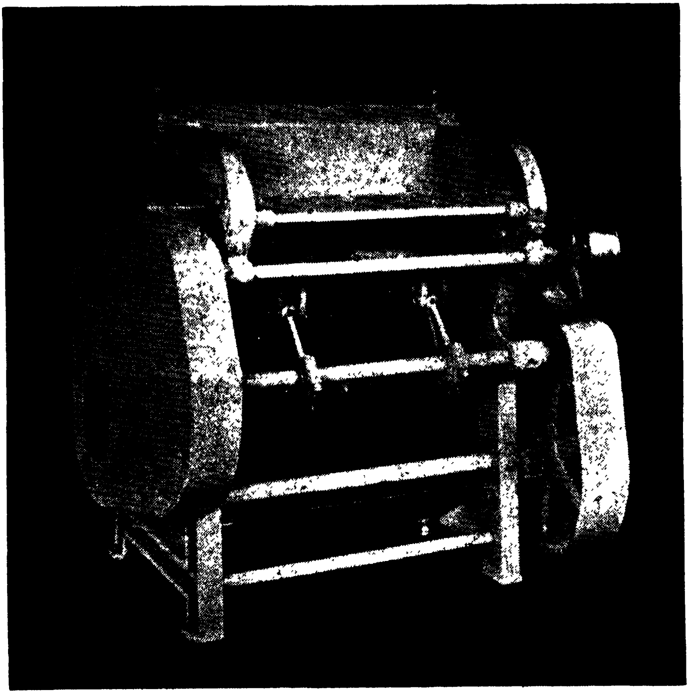

Fig. 20. Casein curd press—continuous type. (*Courtesy of Chemurgic Division, Sheffield Farms Company, Inc., Subsidiary of National Dairy Products Corporation.*)

*Driers.* Since the drying of curd is the most costly item in the manufacture of casein, it is important that the drier be designed and constructed so as to give maximal economy in utilization of power, heat, and labor consistent with the production of casein of acceptable quality. A tunnel drier should be air-tight and the sides, top, bottom, and sliding doors should be well insulated. If two or more driers are required, they may be built so as to have a common wall, without insulation, between them. The tunnel may be constructed of wood, but should be lined preferably with galvanized iron.

The blower should be of the high-speed type to insure circulation of air.

Both blower and heating coils should be oversize, and dampers should be used to regulate the volume of air entering the drier. Manufactures of this equipment can give reliable information on sizes and types required for specific installations.

Drying trays of the standard 30 by 30-inch size can be either purchased or built. They consist of frames of wood or metal with bottoms of No. 14 or 16 galvanized screening. The trucks are heavy wooden frames, 30 by 30 inches, fitted with swivel casters at the corners. The trays, containing 3 to 4 pounds of pressed curd evenly distributed on each, are stacked on the trucks to the height of the drier.

Continuous driers are much more costly than tunnels, but, when they can be used to capacity the year round, are more economical, particularly in respect to labor costs. The casein is usually of better quality, since it is more evenly and rapidly dried than in tunnel driers.

In the conveyor type, the curd is mechanically spread on flexibly connected screens that travel through three compartments, the first and third heated to 130°F, the second to a temperature that may be as high as 200°F. About half an hour is required for the casein to travel through the drier, the speed of travel being regulated to attain the proper degree of dryness.

In the rotary drum type of continuous drier, the curd, fed by a worm conveyor, passes during a period of about three hours downward through a long, slightly inclined, rotating cylinder. Heated air is supplied through steam-heated radiators at the lower, discharge end and passes countercurrent to the curd and out through a suction fan at the feed end. The cylinder is lined with wood or metal and fitted with shelves which, as the drum rotates, are continually lifting the casein and dropping it through the heated air.

Other drying methods that have been advocated are the use of a system of pipes in which a current of hot air supports and dries the curd and carries it, when dry, to the discharge end,[6] and the use of a spray drier.

*Grinders.* Many producers of casein on a small scale sell their product unground. This saves the producer the cost of a grinder and of its operation, but makes necessary more bags for shipping a given weight of casein. The jobber can judge the quality of casein more readily if it is unground and can blend different lots during grinding to insure that the product he sells will be uniform in quality.

Casein grinders are of either the roller type or burr type. The roller-type grinder is usually considered preferable, since the ground particles are more uniform in size than those from the burr grinder, but it is more expensive to buy. A strong magnet should be placed in the feed line to remove pieces of metal that may be in the casein and would injure the grinder or screens if not removed. Mechanically driven, vibrating screens, equipped to return coarse particles to the grinder, are required if the casein is to be sold on a size specification. Most of the casein on the market is ground to pass a 20- to 30-mesh screen, but a few users prefer it ground to pass an 80- to 100-mesh screen.

**Equipment for Continuous Processes.** The equipment required for the Sheffield continuous process[4, 6, 12] is shown in Figure 21. A continuous drier can, of course, be substituted for the tunnel drier shown. The precipitation equipment for the Universal process[14, 15] is shown in Figure 22. Curd presses, curd mills, driers, and grinders used in connection with this process may be of any of the types already discussed.

**Manufacturing Procedures.** *Types of casein.* Caseins may be classified on the basis of the acid used for precipitation as lactic, sulfuric, and muriatic (hydrochloric) caseins. Lactic caseins may be further divided into self-sour and direct-precipitation types. Another classification is based on the type of curd made; hence, pressed-curd, cooked-curd, grain-curd, and continuous-process caseins. Cooked-curd caseins may be further divided into cooked and semi-cooked types. Any one of the three acids mentioned may be used to make curd of any type.

The different characteristics of finished casein depend for the most part on such processing factors as temperature of precipitation, degree of acidity during precipitation and washing, amount of washing, and temperature and time of drying. Caseins of practically the same characteristics can be made by the use of either lactic, sulfuric, or hydrochloric acid, or by the pressed-curd, grain-curd, or continuous-precipitation processes. Cooked-curd casein, however, contains a greater percentage of ash and is less soluble in alkaline solutions than caseins of other types.

*General procedures.* Before discussing in detail the steps, or unit processes, in casein manufacture and the different commercial procedures in use, a brief outline of general procedures and of the reasons underlying them is desirable.

Since casein may properly be considered as in the class of high-grade chemicals, the raw materials should be of high grade and manufacturing procedures should be conducted precisely and uniformly. The skim milk used should be fresh, clean, sweet, and as free of fat as possible, the equipment should be kept clean and sanitary, and the operator should understand the purpose of each step in the procedure, should be capable of making and judging the necessary control tests, and should realize the need for scrupulous care in carrying out each operation.

Casein in milk is united chemically with calcium as calcium caseinate, which is further combined with calcium phosphate, this complex substance

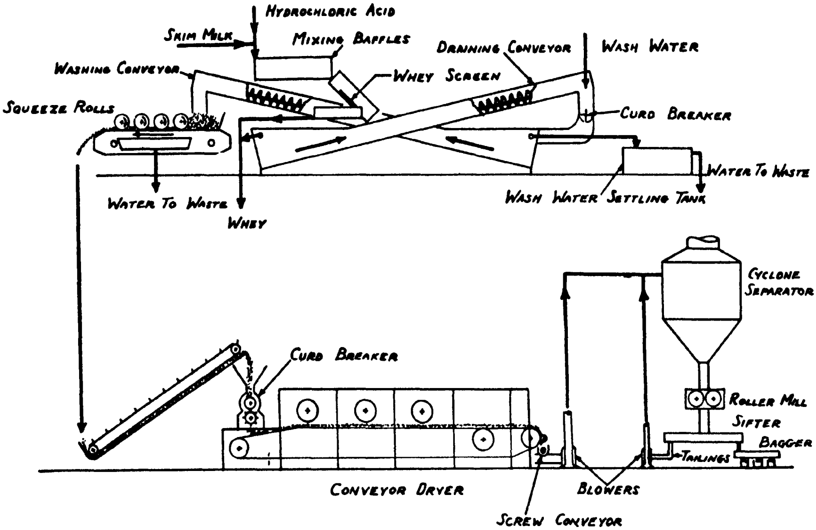

Fig. 21. Process for the manufacture of "New Process" casein. (*Courtesy of Chemurgic Division, Sheffield Farms Company, Inc., Subsidiary of National Dairy Products Corporation.*)

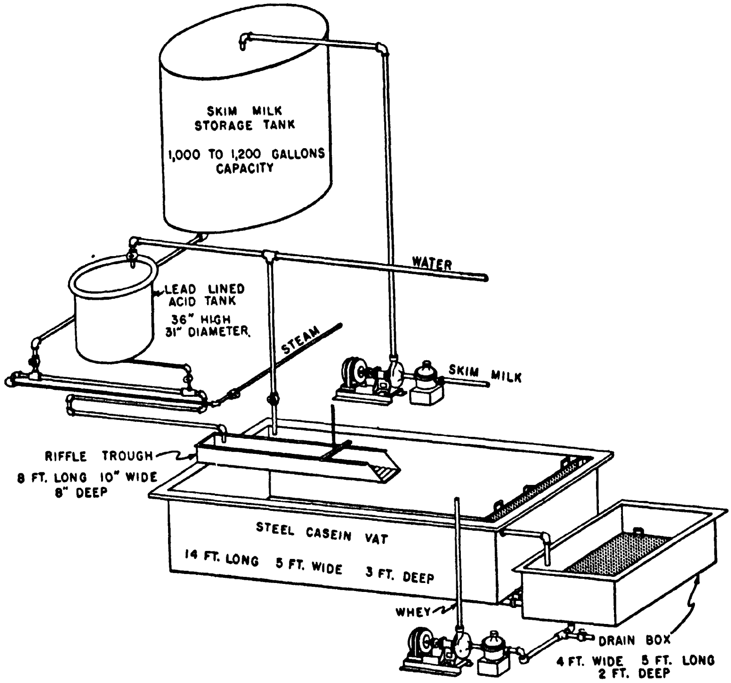

Fig. 22. Process for the manufacture of casein by the "Universal" continuous-precipitation method. (*Courtesy of J. R. Spellacy.*)

being held in colloidal solution at approximately neutrality—actually pH 6.6. If acid is added slowly to milk, casein coagulates as a watery, gel-like curd, which can be made to expel much of its water as whey by adding more acid, or by heating, or by a combination of these two factors. Stirring aids in the expulsion of water. The acid added to the milk forms a soluble compound with the calcium of the caseinate and sets free the calcium phosphate in a soluble form, thus leaving the casein free and practically insoluble. This reaction is sluggish and is speeded both by excess of acid and by heat.

Casein is least soluble at a reaction of approximately pH 4.6, but, once out of solution, it does not dissolve appreciably in either slightly more acid or slightly more alkaline solutions. Consequently, acid may be added until the reaction is at pH 4.1, or even slightly lower, without redissolving casein. This is done in the grain-curd and the continuous-precipitation processes to hasten the solution of calcium and to improve the texture of the curd for ease of washing.

Separation of whey from the curd during draining is incomplete. The whey left in the curd holds salts and other soluble substances in solution. In order to produce a casein as free of salts as possible, it is necessary to wash out this soluble material by stirring the curd with water several times and draining each time, or by spraying the curd with water as in the Sheffield process.

To decrease the cost of drying, pressing is used to remove as much water as is practically possible. Whatever pressing method is used, it is not practical to attempt to reduce the water content to less than about 55 percent before drying.

Pressed curd is shredded to expose as much surface as possible to drying action and thus hasten the drying process. Heat, circulation of air, and, in the rotary drum dryer, agitation of the casein, are the agencies of drying.

Casein is ground in order that as much surface as possible may be exposed to action of the solvent when the casein is being prepared for use. Too fine grinding defeats this purpose, since fine particles have a tendency to form impervious lumps in the presence of solvents.

*Precipitating.* The characteristics of curd are regulated, for the most part, by the degree of acidity and temperature and speed of stirring during precipitation and washing. When acid is being added to skim milk, stirring is necessary to distribute the acid uniformly, but rapid stirring at temperatures below 100°F produces a curd so fine that it settles very slowly during draining and washing and may be lost to some extent in the whey and washings. In self-sour precipitation stirring is needed to break up the curd so that it may settle. Stirring should, in all instances, be at a slow rate.

Temperatures during precipitation should be controlled within narrow limits, since curd is sensitive to heat. A waterjacket is the most satisfactory means of temperature control, but a preheater or live steam may be used. Curd precipitated at temperatures below 95°F is very soft and fine, and, consequently, is slow to settle and difficult to wash without loss. Curd precipitated at temperatures between 95 and 100°F is coarse, provided stirring is not too fast. It can be made firm in either of two ways; it may be heated to a temperature between 100 and 118°F, the exact temperature depending on the acidity; or the acidity may be increased to pH 4.1 without raising the temperature. The latter method produces a granular curd that is easy to drain and wash.

Curd precipitated at about 110°F has what is frequently described as a chewing gum texture; it is stringy, lumpy, and coarse, contains practically no fine particles, and separates sharply from the whey. As such a curd cools to below 110°F, it stiffens and can be easily chopped or broken by agitation into coarse grains that resemble grain-curd in appearance and in ease of draining and washing. This method of precipitation is used in the continuous processes.

Semi-cooked curd, processed at 130 to 135°F, and cooked-curd, processed at 160 to 180°F, are rubbery and plastic and cannot be washed effectively. While still hot, these curds can easily be packed in barrels and hence are usually made when they are to be shipped without further processing. Casein made by a cooked-curd process is hard, tough, difficult to dissolve in the usual solvents, and high in ash and free acid.

The acid necessary for precipitation may be produced by the growth of lactic-acid-producing bacteria in the skim milk, or added as sour whey that has been saved from a previous batch and allowed to develop a high acidity, or by a combination of the two methods. When vat space is ample and the labor schedule permits, the first method is suitable. A vigorously growing whey starter is added to the skim milk, which is kept at 90 to 110°F, preferably close to 100°F. After several hours, sufficient acid will have developed so that on stirring the curd will separate from the whey. If the curd is not sufficiently coarse, time should be allowed for more acid to form or the temperature should be raised. If high-acid-producing lactobacilli are available for a starter culture, whey can be soured to contain 2 percent or more of lactic acid and used for direct precipitation of curd, about one part of whey to three parts of skim milk being sufficient. In the combination of the two methods, the skim milk is inoculated with a starter and allowed to stand until the curd is formed, the whey is then drained off and high-acid whey added to bring the acidity to the point where the texture of the curd becomes suitable for adequate washing.

When sulfuric acid or hydrochloric acid is used to precipitate curd, it should be diluted before being added to the skim milk; otherwise local action of the acid may injure the curd, even though agitation is rapid. High-speed agitation is undesirable because it reduces the size of the curd particles, as has been stated earlier. Within reasonable limits, the more dilute the acid, the better will be the quality of the casein produced. In practice, sulfuric acid is used in dilutions ranging from 1 part in 6 parts water to 1 part in 30; hydrochloric acid in dilutions ranging from 1 part in 3 to 1 part in 9. Earthenware containers should be used in handling and diluting acids and great care should be taken to avoid accidents caused by the heat developed during dilution. If water is poured into sulfuric acid, hot acid will be spattered about and the operator is likely to suffer severe burns. Sulfuric acid should be poured into water slowly with continuous stirring and the water, at the beginning, should be cold. The operator should wear goggles and rubber gloves while handling concentrated acid and while diluting it. Diluted acid should be allowed to cool before being used.

Since, in order to obtain curd of the desired characteristics, it is necessary that temperature and acidity be in proper relationship, both thermometers and means for measuring acidity are needed. The acidity may be determined and expressed in either of two ways—in terms of percentage of acid, or of concentration of effective acid. The percentage of acid is determined by titration with standard alkali and, for convenience, is expressed as percentage of lactic acid, whether the acid is lactic, sulfuric, or hydrochloric. The concentration of effective acid—more precisely, hydrogen-ion concentration—is expressed as pH units. There is only a rough correspondence between percent of acid and pH, because the proportion of acid rendered ineffective by buffer action of the skim milk differs slightly with differences in composition of the milk and somewhat more with the kind of acid used.

Titration equipment adapted for use in making casein may be purchased or may be assembled. A sample of 9 milliliters of whey is titrated to neutrality with 0.1 $N$ alkali; the number of milliliters of alkali required, divided by 10, is the percentage of lactic acid.

To determine pH values, indicator papers, or an indicator solution, and a set of color standards corresponding to several pH values between 4.0 and 5.0 are required. Bromocresol green is the indicator most suitable in this range. Indicator papers are more convenient for use in a plant and are fully as satisfactory as the solution. For use, a paper is dipped in the whey, placed on a window pane not in sunlight, and the color judged immediately against paper standards. With experience, standards become unnecessary. The color of the paper is a deep blue at pH values greater than 5.0 and changes through green to yellow as the acidity increases to pH 4.0. At pH 4.8 the color is bluish green, and at pH 4.1 greenish yellow.

Curd begins to be apparent in skim milk at a temperature of 100°F when the pH value reaches 5.3, which, if the acid is lactic, corresponds to slightly less than 0.5 percent acid. At pH 5.0 to 4.8, or from 0.5 to 0.6 percent acid, the curd is so well defined that, on heating to 104 to 106°F and stirring, it will precipitate, leaving a clear whey. At pH 4.8 to 4.7, or from about 0.55 to 0.65 percent acid, no heating is required, but stirring for 10 to 15 minutes is all that is necessary to obtain complete precipitation and a clear whey. If grain-curd casein is made by the use of hydrochloric acid, the necessary pH value of 4.1 is attained at a titratable acidity of about 0.5 percent. If the acid used is sulfuric and the temperature of precipitation is 120 to 125°F, only about 0.32 percent acidity is required.

*Draining and washing.* It is important that the whey be removed from contact with the curd as soon as possible after precipitation has been completed and the curd has settled, and that the curd be washed immediately. The longer the curd stands in contact with the whey, the more difficult it will be to wash out acid, salts, whey protein, and milk sugar. Poorly washed casein is less soluble than well washed casein, has less adhesive strength, and develops more color during drying.

In draining curd made by a batch process, the curd as it settles should be pushed back from the outlet end of the vat. If the vat was built with the bottom level, it should be blocked up so that the bottom slopes toward the outlet. A coarse wire strainer should be inserted in the vat a foot or more from the outlet to hold back the curd; a board across the vat near the bottom will serve nearly as well. A burlap sack tied over the discharge end of the gate will collect small particles of curd that are carried through in the whey and wash water. As soon as the curd has settled, the valve should be opened wide.

As soon as whey ceases to run freely from the curd, the valve should be closed and wash water added equal in quantity to the whey that has drained off. The water pipe or hose should be of sufficient size—preferably 2 inches in diameter—so that the vat can be filled as rapidly as possible. If the water is alkaline, sufficient acid should be added to it to make it neutral or, especially for the first washing, very slightly acid. Litmus paper may be used to test the reaction of wash waters. The curd should be well stirred in the wash water, either by rakes or by mechanical agitators, but care should be taken not to break the curd into fine particles. The curd should then be allowed to settle and be drained as before. Curd should be washed at least twice, and preferably three or four times.

In the Sheffield continuous process, sprays of water are used for washing, the washings draining in a direction counter to that of the travel of the curd.

*Pressing.* The proportion of water in washed curd and the ease of removal by pressing vary widely with the type of curd made. The percentage of water can usually be reduced by pressing to about 55. Some manufacturers claim to make a curd that requires no pressing, but needs only to be drained for several hours; cooked-curd, on the other hand, is already so low in extraneous water that pressing has little effect on it.

In batch pressing, the washed, drained curd is shoveled from the vat into the press cloths supported in a form. The bottomless, wooden form, 24 inches square and 8 inches deep, is set on the drain board of the press and lined with a press cloth. The curd is then packed into the cloth until the form is full, and the cloth is lapped over the mass of curd. The form is then lifted off, a dividing board placed on top of the wrapped cake of curd, the form placed on the dividing board and lined with a press cloth, and more curd shoveled in. As many cakes are made as the press will hold, care being taken that they are stacked evenly so that pressure will not force them out of line.

When all the curd is in the press, the press screw is tightened to give only a moderate pressure at first, the pressure being increased frequently during the early stages of pressing and only occasionally later. The heavier the springs, the less frequently it will be necessary to tighten the press screw. Pressing is usually begun in the afternoon and is complete the following morning, the cake having been reduced to a thickness of 3 or 4 inches.

As soon as the curd is removed from the press, the forms, drain boards, and cloths should be washed and scalded, and the cloths hung out-of-doors to dry. The press should be sluiced with hot water.

Curd may be pressed continuously in a 2-roll, mechanically driven press, one passage being sufficient to obtain the maximal reduction in water content possible, provided the rolls and rate of feed are properly adjusted.

*Grinding and spreading.* Pressed curd is still liable to deterioration by action of molds and bacteria and therefore should be shredded and dried as promptly as is practicable. The method of operating curd mills needs no description; they are simple machines for producing a mealy porous curd that will dry rapidly and uniformly. If the pressing has not been adequate, grinding will give lumps of curd that will dry on the outside to give a hard, impervious surface that prevents the escape of moisture from the inside, a condition known as case hardening.

The ground curd may be spread on the trays by hand or mechanically.

It should be spread evenly and not more than 2 to 2½ pounds of curd should be placed on each standard tray of 30 by 30 inches. The bottom tray on each truck should be of finer mesh than the others, or should be covered with a cloth, to catch fine particles that may sift through the other trays.

*Drying.* Proper control of temperature and humidity of the air coming in contact with the curd are the essentials of efficient drying of casein. A temperature of 125 to 135°F for the air entering a tunnel drier is suitable for any type of curd. Higher temperatures may be used for well-washed curd, but they are not recommended because of the risk of discoloring the casein and impairing its solubility. Especially during the early stages of drying, it is desirable to recirculate a portion of the air so that the surface of the particles will not become fully dried while the interior is still moist. This partial recirculation effects a saving in heat required, since it shortens the total time required for drying. A suitable rate of supplying air may be calculated by multiplying 4 cubic feet by the number of pounds of wet curd going into the drier each hour. Care should be taken that the temperature in the drier does not rise above 135°F toward the end of the drying when comparatively small amounts of water are being evaporated.

Drying once started should not be interrupted, but should continue until the percentage of moisture is approximately 8 percent. Before removing casein from the drier, large particles and thick layers should be broken and examined to make sure that the interior of the casein is dry. Inadequately pressed casein or casein dried at too high a temperature initially has a tendency to soften and form sheets or lumps in the drying process. Properly dried casein has much the same fine, granular characteristics as the properly ground curd from which it was made.

In the conveyor type of drier, as stated previously, the casein is dried initially at 130°F, then at 200°F, and finally at 130°F. In the rotary drum dryer, air enters at the discharge end at 160 to 170°F and leaves at the feed end at 120 to 130°F.

*Grinding, packing, and storing.* If casein from the drier is to be ground before being stored or shipped, it first should be thoroughly cooled, since warm casein will become plastic in a grinder. In order to produce a uniform product, different batches of casein may be blended and this is most easily done by grinding them together. Most users prefer a casein that will pass a No. 20 or a No. 30 screen, but a few prefer it to be ground to pass a No. 80 or a No. 100. To whatever fineness they are ground, the particles should be as uniform in size as possible. The grinder should, therefore, be adjusted for the size specified,—never for a smaller size,—the casein should be screened after each grinding, and the oversize particles reground.

The dried casein, either ground or unground, is packed in sacks or bags of 100 or 200 pounds capacity. Burlap sacks lined with closely woven cloth or with heavy paper, or heavy three-ply paper bags may be used. The filled containers should be tightly closed and transferred to a clean, dry storage room maintained at a uniform temperature. Wide variations in storage temperatures will cause casein to sweat and subsequently to mold. If casein is stored in a damp atmosphere, it will take up water and will then undergo deterioration. Insects and mice will attack casein, especially ground casein. If casein is to be stored for a considerable time, it is best to store it unground.

The successive steps in the manufacture of acid-precipitated casein having been discussed separately, the most important specific processes in commercial use will be outlined. There are many variations of these processes wherein modifications have been made for reasons arising from local conditions or from special demands. Whether modifications will be advantageous or disadvantageous can be prejudged on the basis of the previous discussion of general procedure and of the effects of various manufacturing details or by means of a test batch.

**Pressed-curd Casein.** The manufacture of pressed-curd casein requires only comparatively inexpensive equipment and is readily adaptable to a varying milk supply. A drier is essential, however, since wet, pressed curd is not suitable for shipping to a drier.

*Self-sour pressed-curd casein.* Only one vat is needed for small-scale operation, the curd being precipitated and washed in the same vat. In larger-scale operation the curd is precipitated in one vat and dropped to a second vat for draining and washing. The temperature of the skim milk is held between 90 and 110°F, direct steam being used for the necessary heating. Five to ten percent of an active starter culture is added and mixed with the milk, and acid allowed to develop to about 0.50 percent, or until the pH value is 4.6 to 4.8. The precipitation is completed by stirring with rakes and allowing more acid to develop or by heating to not over 125°F. As soon as the curd is firm and a sample on settling gives a clear whey, the curd is settled, drained, and washed at least twice with cold water. It is then pressed, dried, and ground.

*Direct-precipitation pressed-curd casein.* Ordinary vats are used for precipitating and washing. The temperature of the skim milk should be 90 too 110°F, when high-acid whey is the precipitant, and 115 to 125°F when sulfuric or hydrochloric acid is used. For the lactic type the milk is stirred and sufficient whey containing about 2 percent lactic acid is added to give an acidity of about 0.50 percent, or a pH value of 4.6 to 4.8, and the precipitation completed by heating to 125°F until the whey is clear. For the sulfuric or hydrochloric acid types, dilute acid is added with stirring until a clean separation of curd and whey is obtained, which will be at an acidity of 0.35 to 0.40 percent acid, or a pH value of about 4.6. Dilute sulfuric acid is prepared by adding one part of commercial acid to at least six parts of cold water—never add water to acid—and cooling before use. Dilute hydrochloric acid is prepared by adding one part of commercial acid to at least three parts of cold water. Earthenware containers should be used for acids.

The curd is settled, drained, washed with cold water two or three times, pressed, dried, and ground. If the curd is sticky, a little acid should be added to the first wash water. It is important that sulfuric acid be as nearly completely washed from curd as possible, since this acid will discolor casein during drying. Sulfuric acid curd has a tendency to be lumpy and should be broken up thoroughly during washing.

**Cooked-curd Casein.** Cooked curd may be shipped in wet condition to a drying plant at some distance; hence, this type of casein can be made in any plant having vats and a steam supply, neither press nor drier being required. The casein made from cooked curd, however, has poor solubility, and is high in ash and free acid. This process is recommended only for dairy plants where pressing and drying equipment are not available. If, instead of the cooking temperature given below, a temperature of about 135°F is used, the product is classed as a semi-cooked casein.

*Self-sour cooked-curd casein.* The vat should be fitted for direct steam and the initial temperature of the skim milk should be 90 to 110°F. Active starter should be added in an amount equal to 5 to 10 percent of the milk, and acid allowed to develop to about 0.50 percent, or until the pH value is about 4.6 to 4.8. Steam is then applied to raise the temperature to 125°F. As soon as the whey is clear, the curd is settled and the whey drawn off. Warm water is added to cover the curd and steam is applied to raise the temperature to 160 to 180°F. The curd will become a plastic, rubber-like mass and, after draining off the water, it should be packed in barrels while still warm.

*Direct-precipitation cooked-curd casein.* The milk should be heated by direct steam to 120 to 125°F and the acid added while the milk is slowly stirred. Whey containing about 2 percent acid is used for the lactic type, and sufficient is added to give an acidity of 0.50 percent acid or a pH value of 4.6 to 4.8. If sulfuric acid is used, it should first be diluted by adding one part commercial acid to six parts water and cooled. The diluted acid should be added slowly to the stirred milk until a clear whey is obtained. This will be at 0.32 to 0.34 percent acid. Similarly, commercial hydrochloric acid may be diluted by adding one part to three parts of water and added to the milk. As soon as the whey is clear, it is drained off, the curd covered with warm water and direct steam applied until the temperature is 160 to 180°F, and the curd has become plastic and rubbery. The water is drained off and the warm curd is packed in barrels for shipment to the drier.

**Grain-curd Casein.** A high-grade casein, low in ash and readily soluble, is made by the grain-curd process, provided pH value and temperature are closely controlled. A jacketed vat is practically essential for accurately controlling temperature. The best product is made by the use of hydrochloric acid, but lactic and sulfuric acids may be used successfully.

*Holding-lactic grain-curd casein.* Warm water is run into the jacket of the vat to hold the skim milk at close to 100°F. Ten percent of an active, high-acid-producing lactic starter is mixed with the milk and acidity allowed to develop to 0.55 percent acid, or preferably 0.65 percent. If the acidity is 0.55 percent, the temperature is raised to 104 to 106°F and the curd stirred until a clear whey separates; if the acidity is 0.65 percent, and the curd is firm, stirring should be continued for 10 to 15 minutes without increasing the temperature. The curd is settled and the whey is drained off as quickly as possible. Speed of draining is more important than completeness. Then whey that contains about 2 percent of acid and has been previously heated to 100°F is added with stirring until the pH value is 4.1. The granular curd is then settled, drained, and washed two or three times with water acidulated to 4.1 and warmed to 90°F, and once with cold, neutral water. About 100 milliliters of concentrated hydrochloric acid per each 1000 pounds of water are required to give a reaction of pH 4.1. The washed curd is pressed, dried and ground.

*Direct-precipitation grain-curd casein.* The temperature of the water in the jacket of the vat should be so regulated as to hold the temperature of the skim milk at close to 100°F for lactic, 95°F for hydrochloric, and 98°F for sulfuric acid curds. The pH value of 4.1 is attained by adding dilute acid slowly during slow stirring of the milk. Approximately 25 percent of whey containing 2 percent of lactic acid is required. If hydrochloric is used, it should be previously diluted by adding one part of commercial acid to nine parts water. If sulfuric is used, one part of commercial acid should be added to thirty parts of cold water and the diluted acid cooled before being used. The curd is settled, drained, washed two or more times with water at 90°F and pH 4.1, and once with cold, neutral water. The washed curd is pressed, dried, and ground.

**Continuous-process Casein.** Two processes involving continuous precipitation and washing of casein are in commercial use—the Sheffield Process, principally in the East, and the Universal Process, principally on the West Coast. Both of these processes are patented. The Sheffield Process normally uses hydrochloric acid as the precipitant, but can use lactic or sulfuric acids. The Universal Process can use any of the usual acids, but high-acid whey, sulfuric acid, or a mixture of the two, are commonly used. Both processes produce a high-quality, high-purity casein.

*Universal Process.*[23, 24] The equipment used in this process is shown in Figure 22. Either diluted sulfuric acid, diluted hydrochloric acid, or sour whey fortified with either of these acids may be used as precipitant. The skim milk and precipitating acid are mixed and the mixture is dropped to the riffle-box. The slope of the riffle-box may be altered to change the size of the curd particles—the steeper the slope, the finer the particles produced. From the riffle-box the curd and whey drop to an inclined, vibrating screen, which allows the whey to pass through and advances the curd to a 2-roll press, whence it enters a beater. There it is broken up sufficiently so that it passes through the perforated bottom to a second inclined, vibrating screen where it is washed by sprays of water. The washed curd enters another 2-roll press and passes through another beater, after which it is ready for drying, either in a tunnel or a continuous drier.

*Sheffield Process.*[5, 9, 21, 22] Equipment for the Sheffield continuous process is shown in Figure 21. The acid tanks, mixer, and baffle box are of stoneware, the chute and conveyor system of acid-resistant metal.

The skim milk is delivered to the mixer at 110°F, being heated by hot water in a concentric-pipe heat-exchanger, thermostatically controlled. Hydrochloric acid, previously diluted to 6 percent concentration, is delivered to the mixer at a carefully controlled rate by way of a constant-level tank and an adjustable cock. The rate of acid feed is adjusted from time to time as necessary on the basis of a titration of the whey or an indicator test. The mixture should be maintained at a pH value of 4.1, which corresponds to approximately 0.5 percent of acid. It travels through a baffle box, in which mixing and precipitation are completed, and thence over a screen that delivers the curd to the conveyor trough at a point near the middle where it is picked up by the worm conveyor. The whey passing through the screen and that flowing down the conveyor trough collect in the sump and overflow. Fine curd that falls to the bottom of the sump is picked up by the worm.

In its travel up the first conveyor, the curd is rolled over, squeezed, and, to some extent, broken into smaller particles. By the time that it has reached the curd breaker, it is well drained and its initial chewing-gum texture has changed into a texture sufficiently friable so that it can be broken into crumbs and pushed through the perforated bottom of the breaker.

The curd then drops to the sump end of the washing conveyor and moves slowly upward, being sprayed by cold water after it emerges from the sump.

The wash water flows counter-current to the direction of travel of the curd, and overflows from the sump to waste.

The washed curd passes to a 2-roll continuous press which is synchronized with the rate of delivery of wet curd. After pressing, the curd is forced through a perforated plate and is then ready for drying either in a tunnel or in a continuous drier. The low-ash, low-acid curd resulting from this process can be dried without discoloration at temperatures somewhat higher than those ordinarily used.

**Yield.** The amount of commercial casein obtained from 100 pounds of skim milk, is on the average, 2.8 pounds, which is equivalent to 1 pound of casein from 35.1 pounds of skim milk. The percentage of casein in skim milk is of the same order and varies only within narrow limits, but this does not signify that all of the casein of the skim milk is obtained as commercial casein. Fine particles of curd are removed in the whey and wash water and some casein is lost during the mechanical processing of the curd and the dried casein, these losses being compensated, however, by the moisture and salts remaining in the finished product.

A yield of 2.6 to 2.9 pounds of high-grade commercial casein per 100 pounds of skim milk should be obtained provided a granular curd is made and washing is thorough. Scanty washing of curd yields a greater weight of product than is obtained when washing is effective, since less casein is lost in the wash water and less ash and other soluble impurities are removed. But, a high-ash casein is a low-grade casein. Maximum yield is desirable from the standpoint of having as much weight of product to sell, but a yield of 3.0 or more pounds per 100 pounds of milk is obtained only by sacrifice of quality. It is logical and fair from the standpoint of both producer and user that a slightly greater price per pound be paid for highest-quality casein than for poor-quality casein to compensate for the slightly lower yield obtained.

**Cost of Manufacture.** Precise figures for costs of manufacture for casein cannot be given, since they vary with costs of labor, water, and steam, with the volume of milk processed, and with other factors such as the value placed on the skim milk used and on the whey produced as a byproduct. The cost of the skim milk is the largest single item, and the cost of drying is next in order of magnitude. Prior to the second world war, some producers were receiving only 5 cents per pound for casein, but this price barely represented costs of production in an area where costs of all items were relatively low. Costs can be calculated precisely only on the basis of local conditions.

**Buttermilk Casein.** A product resembling skim milk casein can be made from buttermilk. However, the protein of buttermilk tends to precipitate in soft, finely divided condition and the fat content of the curd is greater than that of skim milk casein, these factors making the curd difficult to wash without excessive loss and causing the casein to become highly colored during drying. The first difficulty can be overcome to some extent by heating the high-acid buttermilk by passing it through a steam-injector.[8] Another method[14] is to separate the precipitated protein by centrifuging at 300 to 400 times gravity, the protein collecting as a paste on the wall of the centrifuge, from which it may be scraped. The second fault can be avoided by using the casein without drying, as in casein paints, to which use its characteristics are well adapted. A small proportion of buttermilk may be added to skim milk that is to be used in making cooked-curd casein, but the quality of the product is adversely affected.

**Rennet Casein.** The fundamental physical and chemical differences between rennet casein and acid-precipitated casein are not clearly understood, but it can be stated that rennet casein is a calcium caseinate and acid-precipitated casein is free casein—that is, hydrogen caseinate. The two substances differ in solubility and in maximum acid- and base-binding capacity. From the commercial standpoint, acid-precipitated casein is suitable for uses involving adhesive characteristics, rennet casein for manufacture of plastics; neither is well adapted to the purposes for which the other is used.

Rennet curd[26] is precipitated at the pH of fresh milk, 6.6 to 6.8, by addition of rennet extract. A good-grade commercial product contains from 7 to 10 percent moisture and 7.5 percent ash. A less percentage of ash indicates that the milk used in the process had developed acid and had a pH value appreciably lower than pH 6.6; a low-ash rennet casein being essentially a mixture of rennet casein and acid-precipitated casein. A percentage of ash appreciably greater than 7.5 can be attributed to the presence of impurities.

Fresh skim milk is warmed in a water-jacketed vat to 96 to 100°F, and 70 to 100 milliliters of rennet extract previously diluted with 20 times its volume of water is added for each 1000 pounds of milk. This proportion of rennet should cause coagulation to take place in about 20 minutes. If it is found that coagulation occurs in less than 15 minutes, as will be the case if the milk has developed acid, the quantity of rennet used in succeeding batches should be decreased. From 2 to 5 minutes after coagulation begins, as judged best by experience, steam is turned on the water-filled jacket and the curd is cut and stirred until the temperature reaches 150°F.

The curd is then settled and the whey drained through a perforated-bottomed drain rack lined with cloth. The curd is covered with water at about 80°F, thoroughly stirred, settled, and the wash water drained through the drain rack. Two more washings are carried out at 80°F and, on the last, the curd is run to the drain rack. After draining, the curd is pressed for about an hour, shredded, and dried in thin layers in a tunnel drier at a temperature not exceeding 115°F. The dried casein is ground in an impact pulverizer or a roller mill to pass a No. 40 screen and packed in tight bags for shipment.

**Vitamin-free Casein.** There is a steady, though relatively small, demand for vitamin-free casein for use in research on nutrition and in vitamin assays. Long-time extractions with ethanol at 200°F are effective in removing the B vitamins and have been applied to acid-precipitated casein, but such methods are more suitable for small-scale purifications in the laboratory than for employment on a plant scale. Washing with diethyl ether and with acidulated water have also been employed, usually as adjuncts to alcohol treatments.

According to one patented procedure[25] in commercial use, casein is precipitated from skim milk at 200°F by addition of hydrochloric acid to give an acidity of approximately pH 4.6. Vigorous agitation is employed so that the casein will be finely divided. The casein is then pressed or centrifuged. Water equivalent to about two-thirds of the whey removed is heated to 200°F, the casein added, and the mixture stirred vigorously. Sodium chloride equivalent in weight to 2 percent of the water used is added with stirring, and then sufficient hydrochloric acid to bring the pH value of the mixture to between 3.60 and 3.65. The casein is again pressed or centrifuged. The treatment with 2 percent salt solution is repeated. The casein is then given a treatment with 1 percent salt solution, the pH being adjusted to 3.80 to 3.85. It is then given two treatments with water at pH 4.6, dried in a tunnel drier, and ground. This process removes from casein all the known B vitamins, excepting possibly $B_{12}$.

**Uses of Casein.** It should be of practical interest to manufacturers of casein to know the uses made of their product and, through a general knowledge of the processes involved, to understand the requirements of the users relating to specific characteristics of casein. The following descriptions are merely outlines of processes and are not intended to be adequate operating directions.

*Casein plastics.* Rennet casein is now used exclusively for making casein plastics. Earlier, acid-precipitated casein was used, it being dispersed in alkali solutions, coloring matter added, the casein precipitated by addition of acid, pressed into thick sheets in a hydraulic press, and hardened in a formaldehyde bath. After seasoning, the sheets were fabricated into buttons and other items. This so-called "wet process" has been superseded by the "dry process" which uses rennet casein directly without solvent or precipitant.

In the "dry process", rennet casein is added to a heavy-duty mixer and, with agitation running, water is added in quantity sufficient to give a total water content of approximately 40 percent. Dyes are added in solution in the water; pigments are mixed with the casein before the water is added. The mass is mixed till it is uniform in appearance. It should be non-coherent and powdery. It is then fed to a heated, hydraulic press, from which it is extruded as plastic rods at 180°F. The rods are cooled in cold water or, while still soft, are pressed into sheets in a second heated press. The cooled rods or sheets are soaked in a formaldehyde solution for a time sufficient to allow the formaldehyde to penetrate thoroughly, they are then seasoned, and machined to produce the articles desired.

Buttons, costume jewelry, umbrella handles, and many other decorative and useful articles are made from plastic casein. With changes in atmospheric humidity, plastic casein takes up or loses moisture, expanding or contracting; hence, articles in which this characteristic is objectionable are not made from plastic casein.

*Coating of paper.* A thin coating of mineral pigment, such as clay, blanc fixé, or titanium oxide, is applied to paper to produce a smooth, glazed surface on which the finest details of half-tone plates may be reproduced. To cause the particles of pigment to adhere to the paper and to one another, an adhesive is required. Casein is the most satisfactory adhesive substance for this purpose. It not only binds the pigment firmly to the paper, but of itself improves the printing surface and may easily be made waterproof to any degree desired. This last mentioned property is essential in preventing the sticking together of sheets of paper in close contact and in making waterproof wall paper and playing cards.

The casein is softened by soaking it in water which should be cold when added but which may be heated as high as 185°F after addition to the casein. Casein will form a sticky, coherent mass if added to hot water. Coarse casein requires an excessively long time for penetration by water; fine, dusty casein forms lumps in water and penetration is slow. As soon as the casein is thoroughly swelled with water, a solution of an alkali is added. The alkali used is usually borax, ammonia, caustic soda, soda ash, trisodium phosphate, or a mixture of these substances. Only enough alkali is used to dissolve the casein, since excess alkalinity accelerates decomposition of the casein. Slow decomposition is unavoidable and hence the solution is used as promptly as possible after its preparation. The solution is stirred for about an hour at about 130°F to complete the dissolving of the casein, then cooled to below 100°F and added to a mixture of the pigment with water. Coloring materials are added to the coating mixture just before use. The coating is applied by passing the paper through the mixture and spreading it evenly by means of brushes, or by passing the paper between rolls that transfer the coating mixture uniformly to the paper. The paper is then dried in festoons by circulation of heated air.

Some of the defects in coated paper for which the casein may be held responsible are lumps of undissolved casein on the surface, uncoated spots due to foaming of the casein solution, "eyes" supposed to be due to fat in the casein, spots on the paper due to hyphae of molds in the casein, and objectionable odors. The measures that may be taken by the casein manufacturer to prevent these defects will be discussed later in this chapter.

*Casein glue.* Though casein glue may be made in several distinct types, practically all of the commercially used product is a waterproof glue containing casein, caustic soda or an alkaline sodium salt solution as solvent, and calcium hydroxide to render the glue insoluble after application. A dry mixture of the three ingredients may be prepared, which is made ready for use merely by soaking with water, or the casein may be swelled with a small quantity of water, the alkali added in solution in water and mixed with the swollen casein, and finally the calcium hydroxide stirred in. The viscosity of the glue and its working life are two important properties of the glue that are affected by characteristics of the casein used. A large percentage of ash in the casein will cause its solutions to be of relatively high viscosity, a fault that can readily be corrected by addition of more water. The working life of a glue is less, the greater the percentage of ash in the casein. Hence, the glue maker prefers a low-ash casein, but he is likely to be more anxious that the different lots of casein he uses be uniform in ash percentage.

Casein glue has been used widely in glueing plywood, but glues made from synthetic plastic materials are displacing casein glue, since they effect a greater degree of waterproofing of the glued joints.

*Casein paint.* Coatings of widely different compositions and characteristics are included under the name of casein paints. These include whitewashes, containing only lime and skim milk, cold water paints sold both as powders and as pastes for use on exterior and on interior surfaces, and the more recently developed oleocasein and emulsion paints. There is some confusion as to the distinction between oleocasein paints and the emulsion paints, since oleocasein paints are generally oil-in-water emulsions. It is, however, probably most widely accepted that the term emulsion paint should be applied only to that paint in which the casein acts, not as a binder, as in casein paints of other types, but as an emulsifying agent to stabilize the emulsion of the oleoresinous binder in water. Some emulsion paints of the water-in-oil type have been made, but these have not been commercially developed to the stage of the oil-in-water emulsion paints that are thinned with water and are used extensively in interiors on plaster, wall-board, and papered surfaces.

The manufacture of casein paints is highly technical, methods and raw materials are being constantly changed and improved, and information on many details is not available outside the industry. Therefore, no attempt is made here to outline processes of manufacture. Available information may be found in the references at the end of this chapter[17, 26] and in recent patents on this subject.

*Casein fiber.* Commercial production of casein fiber was begun in Italy in 1936 and in the United States about five years later. In 1949 the use of casein for making protein fiber was abandoned in this country in favor of the cheaper vegetable proteins.

The Italian process uses a casein that has been exposed to an acidity of about pH 2.9 during manufacture. It is dissolved in an alkaline solution and the solution allowed to age for several days, whereby the casein is to some degree hydrolyzed and apparently repolymerized, losing sulfur, phosphorus, and some ammonia. Soaps and like ingredients are added and the solution is spun into an acid bath containing salts. The fiber is hardened in formaldehyde solution.

The process employed in the United States made use of an alkaline solution of a high-grade, low-ash casein, with added aluminum salts and soaps. The solution was spun into an acid bath containing salts, and the resulting fiber was stretched, acetylated and hardened. Details of procedure for making artificial fibers from protein can be found in the great number of patents on this subject.

*Casein bristles.*[18] Bristles suitable for use in brushes for applying oil paints are being made commercially from casein. Casein ground to 40–100-mesh fineness is swollen for an hour with approximately 40 percent of its weight of water. The mass is then heated slowly to about 200°F and extruded through dies. The fiber is immersed in a saturated quinone solution and slowly stretched to twice its original length. It is then kept under tension in the quinone solution for 24 hours, during which time it takes up approximately 10 percent of its weight of quinone. It may be further hardened by treatment with 2 percent formaldehyde solution. It is finally washed, and dried under tension.

*Casein films.* Thin transparent sheets consisting principally of casein were made and marketed for wrappings for a short time some years ago. Their manufacture was abandoned because of the unsatisfactory properties of the product. The principal faults experienced appear to have been tendencies for the film to become cloudy and brittle with age. Many patents have been issued on casein films and these should be consulted for details of manufacture. In a typical process a low-ash casein is dissolved in an alkaline solvent, plasticizers, such as castor oil or glycerol, and insolubilizers are added. The film is cast on a base and, after drying, it is stripped. A coating of cellulose ester lacquer may be applied to make the film waterproof.

*Casein in ice cream.* In order to improve the physical characteristics of ice cream, it is desirable to use a mix containing a relatively large percentage of so-called "serum solids," by which is meant milk solids other than fat. If this high percentage of serum solids is obtained by addition of condensed or dried skim milk, the defect known as "sandiness" is likely to appear in the ice cream, a defect attributable to the fact that the concentration of lactose exceeds its solubility and, consequently, the sugar may separate as hard gritty crystals.

Several methods have been developed for obtaining from skim milk products with decreased percentages of lactose, or with no lactose, and for using these products to increase the percentage of serum solids in ice cream. The low-lactose condensed skim milk described in the chapter on Condensed Products is of the first type; preparations derived from acid-precipitated casein are of the second.

It has been found impracticable to use casein directly in ice cream mix, either as curd or as dry casein, because it is insoluble in the mix and imparts an acid flavor to the ice cream.[13] The use of sodium caseinate to replace skim milk has, however, been shown to be possible in amounts up to 5 percent of the serum solids,[4, 28, 32] body, texture and flavor being improved, and overrun increased thereby.

Casein for conversion to sodium caseinate suitable for this purpose should be made from high-quality, pasteurized skim milk. Hydrochloric acid diluted with water in the ratio of 1 to 10 is used as precipitant, and the temperature of the milk during precipitation should be 70°F. The pH value should be brought to 4.6, the milk being stirred during addition of the acid. The curd is washed twice with water adjusted to pH 4.8, and drained in muslin bags until the weight of the curd is between 16.2 and 18.5 pounds per each 100 pounds of milk used.

For the conversion of the wet curd to sodium caseinate, it is transferred to a water-jacketed vat, which should be not over half filled. For each 100 pounds of skim milk used in making the casein, 2 ounces of sodium bicarbonate should be weighed accurately and dissolved in the least amount of warm water possible. The solution, except for a little reserved for later adjustment of pH value, is added to the curd and the mixture stirred vigorously. The water in the jacket of the vat is heated to between 140 and

150°F and the mixture stirred frequently. After about an hour the suspension of sodium caseinate should be of a smooth consistency and contain no curd particles. The pH value is then adjusted to 6.0 to 6.1 with bicarbonate solution and the caseinate put in storage at 32 to 38°F until used.

*Protein hydrolyzates.* Precipitated casein has been utilized in recent years as the starting material in the preparation of mixtures of amino acids to be used nutritionally where rapid and effective utilization of nitrogenous nutrients is required. Whey protein may be used similarly and yields mixtures of less objectionable taste than does casein. Since these mixtures are frequently injected into the blood stream, it is necessary that care be used in their manufacture in order that they may not contain pyrogens, anaphylactogens, or depressor substances. It is also necessary that each batch be assayed to determine whether all the essential amino acids are present and in the desired proportion.

The protein may be hydrolyzed either by means of acid or of enzymes. Acid hydrolysis destroys tryptophane and some other factors; enzyme action does not destroy any of the amino acids, but it is very slow. The following is an example of production by acid hydrolysis. One part of casein is added to 10 parts of 7 *N* sulfuric acid and the mixture heated for 16 hours under 20 pounds pressure in an autoclave. The sulfuric acid is removed by precipitation as the barium salt.

Protein for making hydrolyzates is purchased on specifications, which are essentially the following. The protein should be finely powdered, light brown to white in color, and substantially free from foreign matter and carbohydrates. The ash should be less than 3 percent and should be free from arsenic and heavy metals as shown by U.S.P. tests. The bacteria count should not exceed 30,000 per gram and *E. coli* should be absent. The protein should contain not more than 0.2 percent iron, 0.1 percent calcium, 0.2 percent chloride, 0.3 percent phosphorus, 1.0 percent alkali metals, and 4 percent fat.

*Miscellaneous uses.* The leather industry uses casein as an ingredient of seasonings and pigment finishes. In combination with an alkaline solvent, usually lime, casein is used as a spreader for insecticides, acting not only to aid in coverage, but also to cause the insecticide to adhere to the foliage and to persist in the insoluble film formed as the solution dries. Casein is used to a small extent in textile finishes. Metallic caseinates, such as those of silver and mercury, are used in medicine. Patented therapeutic preparations consisting largely of sodium or ammonium caseinate have had some vogue, mainly in Europe. Many other minor uses, based for the most part on either the adhesive or emulsifying properties of casein, could be listed, but most of them have either been abandoned or never progressed beyond the patent stage.

**Defects in Casein and Their Prevention.** The principal faults attributable to casein by users can usually be detected prior to use by means of the tests and analyses described later, and preventive means based on this information can be taken by the casein manufacturer to avoid these defects in subsequent batches.

*Strong color.* Brown casein is objectionable because its color is imparted to the surface on which it is coated or painted. The cause of objectionable color is usually overheating of the casein during drying, especially in the presence of sulfuric acid or fat. The obvious remedies are using lower drying temperatures, more efficient separation of fat from the skim milk used, and, if sulfuric acid casein is being made, more extensive washing of the curd.

*Poor solubility.* Since most of the uses of casein require that it be dissolved in alkali rapidly and completely, poor solubility is a serious defect. The most frequent causes of poor solubility are high temperatures in cooking curd, high drying temperatures, molding of the curd before drying, and foam on the milk at the time of precipitation of the curd.

*Low strength.* The user objects to a low strength casein because he must use it in higher concentrations than are required if the casein has high adhesive strength. This requires a time-consuming adjustment of his formulas, which is not necessary if the casein is of uniformly high strength. The usual causes of poor strength are high percentage of ash or other impurities, and decomposition of the casein through action of molds or bacteria. Low-strength casein can be avoided by adequate washing, and prompt pressing and drying after washing. In order to improve the efficiency of washing, it may be necessary to improve the texture of the curd by lower temperatures of precipitation, to increase the acidity at precipitation, to wash with slightly acidulated water, and to wash a greater number of times.

*Spots on coated paper.* Spots in the coatings of paper may be caused by the presence of mold spores, dirt, or other insoluble matter, or, in some instances, by excess of fat in the casein. Measures listed above to improve the solubility of the casein, prevention of mold action by prompt and thorough drying of the pressed casein, a thorough cleaning of the walls, floor and equipment of the plant, and the screening of the air intake of the drier are means of preventing this defect. The "fish-eyes," usually attributed to excess fat in the casein, can be avoided by improving the efficiency of separation of cream from the milk used in making the casein.

*Uncoated spots on coated paper.* The cause of this defect is practically always foaming of the coating mixture. Caseins precipitated at temperatures below 95°F and with rapid agitation are especially likely to give solutions that foam. Avoidance of low temperatures and high speed agitation during precipitation of curd will usually remedy this defect. Blending of several lots of casein, including a non-foaming casein, is an effective method of reducing foaming, since small proportions of non-foaming caseins are usually remarkably effective in reducing the foaming tendencies of other caseins with which they are mixed.

**Specifications.** No generally accepted specifications have been established for commercial casein and the specifications of users differ in many respects. Reasonable requirements for rennet casein include that it be tasteless, have only a faint, agreeable odor, be not darker than light yellow or cream in color, have a pH value of approximately 7.0, contain not more than 10.0 percent moisture, and have approximately 7.5 percent ash. Acid-precipitated casein, to be generally acceptable, should have only a faint pleasant odor, should be light cream or nearly white in color, should have total acidity requiring not more than 10.5 milliliters of 0.10 $N$ alkali per gram, and should contain not more than 10.0 percent moisture, not more than 1.0 percent fat (on a moisture-free basis), not more than 4.0 percent ash (on a moisture-free basis), and not less than 14.25 percent nitrogen (on a moisture-, fat-, and ash-free basis). Specifications of strength and solubility vary with the purpose for which the casein is to be used.

**Analysis and Testing of Casein.** In order to know whether any given lot of casein will satisfy the specifications of a user, it is necessary to apply certain physical and chemical tests. Not all of the tests and analyses described here should be required in any one instance, since some duplication of the information given is involved. All tests that are likely to be required are given.

*Color.* The color of commercial casein may be nearly white, reddish brown, or any shade between. Strongly colored, unground caseins become much lighter on grinding. Most of the high-grade ground casein on the market is a very pale yellow or cream in color. No color standards have been established for casein, although this has been done for similar products.

*Odor.* Dry casein should have only a faint inoffensive odor. Unpleasant odors may appear when casein is dissolved in alkaline solutions; hence, odor tests are usually made on such solutions. For a suitable test, 10 grams of casein is soaked in about 10 milliliters of water and an equal volume of thick milk of lime is added with stirring. The mixture is allowed to stand for a few minutes and the odor noted. The odor should be slight and not unpleasant.

*Cleanliness, mechanical.* It is obviously difficult to set up a standard for the cleanliness of casein or to express quantitatively the amount of dirt in a sample of casein. Visual inspection will detect such foreign material as bits of metal and cloth and splinters of wood. More information may be obtained by the aid of a hand lens or a low-power microscope. Another method consists of preparing a dilute solution of casein, allowing it to stand for an hour, and examining the solution for foreign material that may either float on the surface or sink to the bottom. A water solution containing 0.5 percent sodium hydroxide and 5 percent casein is suitable for this purpose.

*Cleanliness, biological.* Beetles and moths are readily detected by visual examination and molds may also be visible in instances of gross contamination. The number of bacteria, yeasts, and molds present in casein may be determined by plate count, or information of similar nature may be obtained by the Warth incubation test.[26]

Microbiological contamination becomes of interest when casein is to be stored under conditions in which there is danger of condensation of moisture on the casein because of high humidity or variable temperature. Since all commercial caseins probably contain some microorganisms and readily become inoculated with them subsequent to manufacture, the count of organisms in freshly-made casein is information that soon becomes obsolete. Odor, solubility, moisture, and strength tests are usually adequate to detect defects in which the activities of microorganisms have played a part.

*Fineness.* Nests of standard screens that can be shaken mechanically may be purchased for use in determining fineness of casein. A known weight of casein is shaken for a definite time and the weights of the fractions remaining on each screen are calculated as percentages of the sample taken. Hand screening is tedious and the results are less precise than those obtained by means of mechanically shaken screens. A 50-gram sample is placed on a screen of known mesh-size and shaken for 10 minutes in a horizontal plane at a rate of approximately 120 strokes per minute, the screen being allowed, at the end of each stroke, to strike the palm of the free hand, which is held stationary. The portion passing through the screen is calculated as percentage of the sample taken, and reported as the percentage passing the screen employed.

*Solubility.* Water solutions are prepared containing 24, 30, and 40 grams of borax ($Na_2B_4O_7 \cdot 10H_2O$) per liter and these are labeled "12-percent borax," "15-percent borax," and "20-percent borax," respectively. Fifty milliliters of the "12-percent borax" is added to 10 grams casein that has been ground to pass a No. 20 screen. This gives a mixture containing 1.2 grams borax and 10 grams casein, the quantity of borax being 12 percent of that of the casein. The mixture is stirred and placed in a water bath held at 149°F. It is stirred continuously for 5 minutes, then at the end of 10 minutes, and again at the end of 15 minutes. If the casein dissolves completely, it may be reported as "soluble in 12-percent borax;" otherwise the test should be repeated on another 10-gram sample of casein with 50 milliliters of the "15-percent borax." If this does not give complete solution, the test is carried out with the "20-percent borax." Caseins insoluble in "20-percent borax" are considered of very poor quality. Frequently the sample will dissolve completely except for a few particles. These insoluble particles are usually supposed to be attributable to foam on the skim milk during the precipitation of the casein.

*Strength.* Any test to show the adhesive strength of casein should be made under conditions approximating those under which the casein is to be used. Since methods of use of casein differ widely, no test gives information adequate for all users. The test described here gives comparative strength values for casein as most generally used in the coating of paper, and is sufficiently precise to satisfy the needs of most producers of casein. More precise results can be obtained by making the tests under standard conditions of humidity, and additional information of interest to the user of coated paper can be obtained by using a series of waxes of graded adhesive characteristics to pull the coatings from the paper.

Twenty grams of casein are weighed into a tared beaker containing a stirring rod. About 70 milliliters of water is added to the casein, and the mixture is allowed to stand for half an hour with frequent stirring. Then 2.4 grams of powdered chemically pure borax is added, and the mixture warmed and stirred until solution is complete. Some caseins may require more borax to dissolve completely. It is essential that the mixture shall turn a drop of an alcohol solution of phenolphthalein pink. When solution is complete, enough water is added to make the total weight of the solution 100 grams. Each 5 grams of the solution then contains 1 gram of casein.

One hundred grams of dried clay is weighed into a casserole supplied with a blunt rod for crushing lumps and mixing. A standard clay should be used; that sold under the designation "Kaolin, Special for Tannin" has been found satisfactory. Seventy milliliters of water is added and the whole mixed to a smooth paste. The casserole and contents are then counterbalanced and 30 grams of the casein solution weighed in. After thorough mixing, a small amount is spread on a sheet of paper by means of a straight-edged brass scraper or a camel's-hair brush. The sheet is marked "6" to denote the number of grams of casein per 100 grams of clay, and put aside to dry. The casserole is again counterbalanced and 5 grams more of the casein solution weighed in. The mixing and coating are repeated, and this sheet is marked "7." Further increments of 5 grams of casein are added, and coatings made of 8, 9, 10, 11, and 12 grams casein per 100 grams clay.

After the coated papers have become thoroughly dry, areas of uniform thickness of coating are selected by holding them to the light, and these areas are marked. Sticks of good-quality sealing wax are well softened in a flame and pressed on the selected areas. After the wax has cooled for 30 minutes, the fingers of one hand are placed on the paper beside the wax, and the stick lifted with a steady pull by the other hand. If the coating comes away without pulling any of the fibers of the paper, the test is negative. If the whole surface of the paper is pulled away, the test is positive. If part of the fibers are torn away, the test is considered on the border line.

High-grade caseins will give positive tests on the sheets marked "7," "8," or "9"; caseins that do not pull the surface of the paper until the sheets marked "11" or "12" are reached are clearly inferior in strength.

Since the results are affected by differences in the clay and in the paper used, it is important that the same grade of clay and type of paper be used consistently. The quantities of all substances used in the coating solutions may be halved, provided the quantity of solution used for each coating is kept small.

*Viscosity.* The viscosities of casein solutions differ, not only with different caseins, but with the concentration, pH value, temperature and age of the solution. Consequently, it is necessary to standardize as many of these factors as possible to obtain reproducible and comparable results. Since, for most purposes, only comparative values are required, a simple method of comparing times of flow of casein solutions from the same orifice and under the same head is often adequate. For determinations in absolute units, the viscosimeters commercially available may be employed, or a buret consistometer[3, 15] may be constructed and calibrated for the purpose.

The following procedure has been found satisfactory for preparing casein solutions for viscosity determinations.[1] For each 100 milliliters of solution required, 9 grams of casein are swelled with a little water and dissolved in 0.1 $N$ sodium hydroxide. The solution is held at 50°F over night, allowed to warm to 86°F in the forenoon of the following day, the pH value then adjusted to between 9.1 and 9.2, the volume adjusted to 100 milliliters, and the viscosity measured at 86°F in the afternoon.

*Foaming tendency.* Foaming of clay-casein slips during application to paper is probably the most troublesome difficulty experienced in paper coating. It can be considerably decreased by adding antifoaming agents to the slip, but, since the chief causes seem to be the precipitation of the casein at temperatures below 90°F and precipitation with too rapid agitation, the most effective remedy can be applied in the casein plant.

The method described below for measuring foaming tendency of casein is essentially that devised by Richards and McFail,[19] and gives relative values. Different stirring assemblies will give different values on the same sample of casein.

A 110-volt, 60 cycle, alternating-current, $\frac{1}{15}$-horse-power, synchronous motor mounted with its shaft vertical, and operating at a speed of 1,800 revolutions per minute, is used. The stirrer is mounted on the end of a round shaft $\frac{1}{4}$-inch in diameter and 4 inches long, and consists of a blade $1\frac{3}{8}$-inches long horizontally, $\frac{1}{4}$-inch wide vertically, and $\frac{1}{32}$-inch thick. The stirrer and shaft are plated with nickel.

The slips are prepared by softening 11 grams of casein in 55 milliliters of water, adding $\frac{1}{2}$-gram of soda ash, heating the solution to 122°F, and mixing with a suspension of 100 grams of clay ("Kaolin, Special for Tannin") in 100 milliliters of water. The dry clay is sifted through a No. 40 screen before use.

One hundred milliliters of the slip is poured into a 100-milliliter graduate, and the graduate and contents are weighed. Two hundred grams of the slip is then placed in a 400-milliliter, low-form beaker, the motordriven stirrer placed so that the top of the blade is $\frac{3}{4}$-inch below the surface and the stirrer run for 5 minutes. After standing for 1 minute, 100 milliliters of the slip is poured from the beaker into the same 100-milliliter graduate as before, and the graduate and contents are weighed. The difference between the weights of 100 milliliters before and after being stirred is a measure of the foaming tendency and is called the foaming index.

*Nitrogen.* A 1-gram sample of casein is weighed into a Kjeldahl digestion flask, and approximately 0.7-gram of mercuric oxide, 0.2-gram of copper sulfate crystals, and 25 milliliters of nitrogen-free, concentrated sulfuric acid added. The contents of the flask are heated over a low flame until frothing ceases, and then boiled until the mixture is clear and for a short time thereafter, or about 2 hours altogether. The mixture is cooled and diluted with 200 milliliters of water. A few pieces of granulated zinc and 25 milliliters of 40-percent sodium sulfide solution are added. Without mixing, about 50 milliliters of 40-percent sodium hydroxide is poured slowly down the neck of the flask, and the flask connected immediately with a Kjeldahl distillation unit. After shaking the flask to mix the contents, the freed ammonia is distilled into 25 milliliters of 0.5 $N$ acid. As soon as about 150 milliliters of water has distilled, the excess acid in the receiver is titrated with 0.1 $N$ sodium hydroxide. Percent nitrogen equals 0.14 (125 − ml. 0.1 $N$ NaOH required).

Pure, dry casein contains 15.51 percent nitrogen. Commercial casein should contain not less than 14.25 percent nitrogen on a fat-, ash-, and moisture-free basis. This percentage multiplied by the usual protein/nitrogen factor of 6.38 gives 91 percent protein. However, since commercial casein may contain small amounts of nitrogenous material other than casein, a percentage thus calculated should not be assumed to represent only casein.

*Moisture.* Approximately 1 gram of casein is weighed accurately into a tared silica or platinum dish, and heated at 208.5°F for 5 hours in a vacuum oven. The sample is then cooled in a desiccator and weighed. It is then heated in the oven for 1 hour, cooled and weighed, and this is repeated until the weight is constant. The percentage of moisture is calculated from the loss of weight.

*Ash.* The dry sample from the moisture determination may be used for the determination of ash. If the sample contains less than 2.5 percent ash, it is necessary to add calcium in the form of an organic salt, such as the acetate, to prevent volatilization of organic phosphorus during ashing. Two milliliters of 0.1 molal calcium acetate solution, equivalent to 0.0122 gram calcium oxide, is added to the 1-gram sample of casein and the sample is charred over a microburner, without its being allowed to burst into flame. When charring is complete, the dish is allowed to cool, and 1 or 2 milliliters of ash-free nitric acid is added. The acid is evaporated on an electric hot plate or over a small flame, and the dish placed in a muffle at 1100 to 1200°F, or heated over a flame to redness, until the ash is white. The dish is cooled in a desiccator and weighed. The weight of calcium oxide derived from the calcium acetate added is subtracted from the weight of the ashed sample before calculating the percentage of ash.

It should be noted that pure, dry casein contains phosphorus sufficient to give an ash percentage of 1.80 and that ash percentages less than 1.80 indicate loss of organic phosphorus during ashing.

*Total acidity.* What is sought by determination of total acidity is a measure of the quantity of acid that must be neutralized in preparing a solution of casein. Because the reaction of casein with alkalies is slow and because the technique of the determination does not parallel commercial practice in preparing casein solutions, the results obtained must be considered empirical and of value only as a useful basis for comparing alkali consumption by different caseins.

A 1-gram sample of casein is weighed into a flask and, with gentle shaking of the flask, 25 milliliters of 0.1 $N$ sodium hydroxide is added from a pipet. The flask is then stoppered and shaken until the casein is dissolved. The stopper is then removed and adhering solution washed into the flask by means of a stream of water from a wash bottle. One hundred milliliters of distilled water and 0.5 milliliters of a 1 percent solution of phenolphthalein in alcohol are added, and the solution titrated with 0.1 $N$ acid. The acid should be added rapidly with constant shaking of the flask to prevent local precipitation of curd. The number of milliliters of 0.1 $N$ alkali used minus the number of milliliters of 0.1 $N$ acid required is the total acidity. Directions must be followed precisely in order to obtain concordant results. Values are usually calculated to the basis of 1 gram of fat-, moisture-, and ash-free casein.

Pure, dry isoelectric casein should require 9.0 milliliters of 0.1 $N$ alkali per gram by this test. Commercial caseins consume from 6 to 14.

*Free acidity.* The determination of free acidity is a measure of the effectiveness of washing of the curd during the manufacture of casein. Since this determination is made on a water extract of the casein, the value obtained depends, not only on the free acid present, but also on the soluble buffer substances in the casein.

Ten grams of 20-mesh casein is mixed thoroughly with 100 milliliters of water, and the mixture allowed to stand for an hour with occasional stirring. Fifty milliliters of the supernatant liquid is then transferred to another flask by means of a pipet and titrated with 0.1 $N$ sodium hydroxide, phenolphthalein solution being used as indicator. The solution should be stirred while it is being titrated, and the alkali should be added slowly till a pink color persists throughout the solution. The free-acid value is expressed as the number of milliliters of 0.1 $N$ alkali required in the titration. An effectively washed casein will require less than 4 milliliters of alkali.

*pH value.* The reaction of a 5 percent suspension of casein in water may be determined by means of the usual electrodes used for pH determination. The suspension should be allowed to stand for an hour with frequent shaking before the determination is made. A casein precipitated at a pH value below 4.6 and washed thoroughly will have a pH value approaching 4.6, but a casein precipitated at pH 4.6 will have that value whether washed well or not. Consequently, this determination is not always informative, if considered alone.

*Fat.* Of the methods published for the determination of fat in casein, those in which the casein is dissolved in alkali and the solution extracted with ether give high results because substances other than fat are extracted. The method of direct extraction of dried casein with ether and the modified Babcock method give results that are in good agreement with each other. Since the Babcock method is much more rapid than the direct extraction, it is generally preferred and is the method chosen to be described here.

Two grams of casein is soaked in 6 milliliters of water in a small beaker for about ½ hour; then, with constant stirring, 9 milliliters of concentrated sulfuric acid—specific gravity, 1.84—is added. The mixture is poured into a Babcock skim-milk test bottle, the beaker is rinsed with 5 milliliters of water and 5 milliliters of sulfuric acid and the washings are poured into the test bottle. The bottle is filled to the base of the neck with sulfuric acid that has been diluted in the proportion of 5 volumes of acid to 4 volumes of water, placed in the centrifuge and whirled for 5 minutes. It is then filled nearly to the top of the graduated portion of the neck with the diluted acid, whirled for 2 minutes, and the length of the fat column read immediately. The reading multiplied by 9 gives the percentage of fat in the casein.

*Milk sugar.* Ten grams of casein are weighed into a 500-milliliter, wide-mouth bottle and a mixture of 120 milliliters of water and 130 milliliters of 95 percent ethyl alcohol is added. The bottle is tightly stoppered and shaken for four hours in a motor-driven shaking machine. The mixture is allowed to settle for several hours and the supernatant liquid decanted. If the solution is turbid, it is centrifuged. Ten milliliters are removed by means of a pipet and milk sugar is determined by any one of the methods for reducing sugars.

The percentage of milk sugar in casein is an index to the effectiveness of washing. Since the ash determination gives practically the same information, milk sugar is not often determined. An unwashed casein may contain as much as 5 percent of milk sugar, a well-washed casein only a fraction of a percent.

*Calcium.* Two grams of casein are weighed into a crucible and heated carefully until charred. The residue is warmed with a few milliliters of dilute hydrochloric acid, the mixture filtered, and the filter washed several times. The filter paper and contents are dried and ignited in a crucible until the residue is white, the digestion with hydrochloric acid, filtration and washing repeated, and the combined filtrates and washings diluted with water to a volume of 100 milliliters in a graduated flask. To an aliquot of 50 milliliters in a beaker is added 1 milliliter of concentrated sulfuric acid and the mixture heated nearly to boiling, one hundred milliliters of 95 percent alcohol is added, the mixture stirred, and allowed to stand over night. The precipitate is collected on a weighed Gooch crucible, washed with alcohol until free of acid, dried, gently ignited and weighed as calcium sulfate.

*Phosphorus.* The ash percentage obtained in the determination of ash percentage may be used for the determination of phosphorus. It is necessary that sufficient calcium acetate to prevent volatilization of phosphorus shall have been added to low-ash caseins; otherwise, results will be too low and not reproducible. Five to 10 milliliters of concentrated nitric acid is added to the crucible containing the ash, and the mixture heated on a steam bath. The solution is washed into a beaker with a little water, and boiled. It is then cooled, carefully neutralized with ammonia solution, and made barely acid with nitric acid. Two grams of solid ammonium nitrate is added, the solution heated nearly to boiling, and ammonium molybdate solution added in excess to precipitate the phosphorus. The solution is then heated at 149°F, for an hour. A few drops of ammonium molybdate solution is added to make sure that precipitation is complete, the precipitate is separated by filtration, and washed several times with water containing 5 percent ammonium nitrate. The precipitate is then dissolved by means of hot ammonia solution, and the filter paper washed with hot water until the volume of the filtrate and washings is slightly less than 100 milliliters. The solution is nearly neutralized with hydrochloric acid and cooled. With constant stirring, magnesia mixture (prepared by dissolving 55 grams magnesium chloride and 70 grams ammonium chloride in 650 milliliters of water and diluting the solution to 1 liter with 10 percent ammonia solution) is added in excess to precipitate the phosphorus as magnesium ammonium phosphate. After about 15 minutes, 12 milliliters of concentrated ammonia solution is added, and the mixture allowed to stand over night. The precipitate is collected on a Gooch crucible, washed with dilute ammonia solution until the washings are free of chlorides, dried, and ignited to constant weight as magnesium pyrophosphate, $Mg_2P_2O_7$, from which the percentage of phosphorus in the sample may be calculated.

## WHEY PROTEIN

Whey protein has been considered of minor practical importance in the consideration of utilization of milk byproducts, largely because of the relatively small quantities present in skim milk, buttermilk and whey. There is usually no adequate reason for removing whey protein from milk products that are destined to be used in food or feed, for its nutritional value is high. However, there is more and more objection being received to the dumping of whey into streams and sewers and, since the whey protein accounts for about one-half of the oxygen demand of whey, the removal and utilization of the whey protein is a partial solution of the problem of whey disposal. In the manufacture of lactose and lactic acid from whey, however, it is necessary to remove the whey protein. This is usually accomplished by coagulating it by heat and isolating it in an impure, denatured form which is used for animal feed.

In the Scandinavian countries cheeses are made from whey, either by evaporation of water until the mass is of the desired consistency, or by coagulating the whey protein, collecting it and pressing it in hoops. In the United States, concentrated or dried whey is added to processed cheese and cheese spreads by some manufacturers. Whey cheese, condensed whey, and dried whey are discussed elsewhere in this book.

Whey protein for nutritional uses, such as in infant foods, may be produced either as a heat-denatured substance insoluble in water, as a soluble sodium or ammonium salt prepared from the denatured form or as the undenatured protein. Whey protein may be converted to a mixture of amino acids for nutritional use, by methods described earlier in this chapter, for the production of hydrolyzates from casein.

It is sometimes desired to recover separately, for nutritional use, the calcium phosphate present in whey. This may be accomplished[2, 20, 26] prior to coagulation of the protein by adjusting the pH of the whey first to about 4.5 and then to 6.2. The precipitate, which consists of calcium phosphate and some protein, is removed by filtration and the filtrate treated for isolation of the whey protein by one of the processes described in the following paragraphs.

**Denatured Whey Protein.** Methods of isolation of denatured whey protein as a byproduct in the production of lactose and of lactic acid are described in Chapters 11 and 3, respectively. If it is desired to produce a denatured whey protein of relatively high purity and in maximum yield the procedure described here is more suitable.[11] The reaction of the whey is brought within the range of pH 4.35 to 4.85, preferably 4.75, by addition of acid or alkali, and the whey heated to a temperature well above 160°F until flocculation is complete. The whey protein is then separated by filtration, washed, and either dried or converted into soluble protein by agitation with a solution of sodium or ammonium hydroxide, carbonate or bicarbonate of sufficient concentration so that the final reaction will be between pH 6.40 and 6.99. If a more highly pure protein is desired, the solution may be acidified to pH 4.57 and the reprecipitated protein treated as described above. The solution of protein is dried, preferably by means of a drum or spray drier.

Another procedure for preparing a soluble denatured whey protein[1] is to mix the paste of separated protein with 2 percent of its dry weight of powdered sodium hydroxide and dry the mixture in a vacuum shelf drier.

The yield of whey protein should be greater than 50 percent of the total nitrogenous constituents of the whey.

**Undenatured Whey Protein.** In order to avoid denaturation, it is necessary that whey protein not be subjected to temperatures above 150°F for an extended time. Consequently, use is made of precipitants and of vacuum evaporation and drying.

In a process[30] that may be conducted as an adjunct to the manufacture of lactose, whey is neutralized and evaporated in a vacuum pan to a concentration such that the greater part of the lactose crystallizes. Detailed directions for this part of the procedure will be found in the chapter on lactose. After the crystallized lactose is removed by centrifuging, the filtrate may be dialyzed to remove salts and more of the lactose.[29] The filtrate is evaporated further under vacuum and dried at a low temperature, preferably in a vacuum drier. The whey protein thus obtained contains salts and lactose, the proportions depending on the extent of the dialysis.

In a process[16] based on the insolubility of whey protein in ethyl alcohol, one part of whey powder is thoroughly mixed with 20 parts of ethyl alcohol of at least 71 percent concentration, and the mixture filtered promptly. The filter cake is dried in a vacuum shelf drier and contains about 50 percent of soluble whey protein. The filtrate may be utilized for the production of lactose and a riboflavin concentrate. This process is described in more detail in the chapter on lactose.

In another process,[12] whey protein is precipitated from whey by addition of sodium metaphosphate at a pH value of about 3.0. The precipitate is collected by filtration, washed, and then treated with a sufficient excess of milk of lime to give a pH value of about 9.0. This operation converts the metaphosphate into an insoluble salt and at the same time dissolves the protein. After filtration, the filtrate is acidified to pH 5.0 to cause flocculation of the protein. After complete precipitation, the suspension is brought to pH 7.0 by addition of alkali and centrifuged. The protein is dried at 105 to 120°F under 20 to 40 millimeters pressure. It is relatively pure, undenatured, and suitable for use in foods.

Another procedure[10] that appears to be practical is that of freezing out water from whey and precipitating the protein from the concentrate by addition of methyl alcohol.

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# 11

# LACTOSE

## PRODUCTION

The annual production of lactose in the United States in the two decades immediately preceding World War II varied between 3 and 6 million pounds, the average being slightly more than 4 million pounds. Because of the demand for lactose to be used in the manufacture of penicillin during the war, production increased until nearly 13 million pounds was made during 1944.

## SOURCE

Whey from the manufacture of casein precipitated by means of hydrochloric acid has been the favorite source of lactose, but increased demands have made it necessary to use cheese whey. Since 300 to 400 million pounds of lactose are potentially available each year in the whey produced in this country, it is evident that the lactose isolated from whey represents less than 5 percent of what it would be possible to produce if there were unlimited demand.

Though most processes in commercial use for making lactose start with fluid whey, one process, which will be described, has been developed on a laboratory scale for using dried cheese whey. A process that has been employed commercially to a slight extent for making low-lactose skim milk for use in ice cream, produces lactose as a byproduct.[10, 11, 28] This is described in Chapter 4.

Several other methods have been advocated, such as the drying of whey, followed by fractional extraction with water;[7, 9] the freezing out of water from whey, followed by either crystallization of the lactose at 32°F,[4] or extraction with alcohol;[6] and the drying of clarified whey.[25] Many patents have been issued for special methods of clarification of whey prior to separation of lactose.

## USES

Refined lactose has been used mostly in foods for infants and invalids and in solid pharmaceutical preparations, such as pills and tablets, in which it serves as an inert diluent and suitable base for drugs. Other uses, accounting for only small quantities of lactose, are in silvering mirrors, for preserving oilcake and rubber latex, and for giving a crystalline, frosty appearance to bottled liqueurs. The extensive use of lactose in the manufacture of penicillin is attributed to its slow rate of fermentation and resulting effect in preventing the destruction of the penicillin after its formation and requires at present more lactose than all other uses combined. How long the demand for lactose for this purpose will continue cannot be predicted, because of the likelihood that penicillin, or adequate substitutes, will be produced by synthetic procedures that will make the currently used process obsolete.

Lactose may be used as a source of fermentation products, such as lactic acid, but it is neither necessary nor economical to isolate the lactose from whey prior to carrying out fermentation procedures. Fermentation products from lactose are discussed in Chapter 3.

## SCALE OF MANUFACTURE

The manufacture of lactose, in order to be profitable, should be conducted on a scale requiring a minimum of about 100,000 pounds of whey per day. From 2000 to 3000 pounds of lactose can be produced from this quantity of whey. If the manufacture of lactose is a part of more extensive dairy product manufacture and is closely integrated with it, sharing labor, equipment and power, smaller scale operation may be practical.

## EQUIPMENT

The equipment needed in lactose manufacture is shown in the accompanying flowsheets,[20, 21] some of it being equipment commonly used in dairy plants. The tunnel drier is of the type used in casein manufacture and described in Chapter 10. The vacuum evaporator may be the same as is used in condensing milk, but the filter press, disk filter and sugar centrifugal are items not employed in the usual dairy plant operations.

## GRADES OF LACTOSE

Lactose may be considered as of three grades: crude, technical and U.S.P. Crude lactose is the product of the first stage of the common process of manufacture. It is obtained by crystallization of the lactose from concentrated whey, no procedures other than partial removal of protein and washing of the sugar crystals having been employed to reduce the percentages of protein and ash. It contains more than 2 percent of protein plus ash, and is used ordinarily only for refining to U.S.P. lactose, though, if made under sanitary conditions, it is suitable for use in infant foods and in fermentation processes. It gradually develops an objectionable odor in storage because of the protein present. Technical lactose contains less than 1 percent of protein plus ash, but is not sufficiently pure to meet the standards of the United States Pharmacopoeia for refined lactose. Technical lactose may be made by processes that include only one crystallization, but employ special means for removal of protein. This grade is suitable for use in infant foods, in fermentation media, and in making the explosive lactose nitrate. U.S.P. lactose has been refined by recrystallization and meets the specifications of the United States Pharmacopoeia.[15] This is the grade used in pharmaceutical preparations and sold in drug stores for use in preparing formulas for infant feeding.

## DIFFICULTIES IN THE ISOLATION OF LACTOSE

The degree to which protein and salts are removed from whey prior to its concentration for crystallization of lactose largely determines the purity of the lactose. It is obvious that protein and salts present during crystallization will contaminate the sugar, especially if they have precipitated during the concentration of the whey. The presence of these substances in solution may give rise to more troublesome difficulties, however, since it may greatly increase the viscosity of the concentrated whey, thus making the separation of the crystallized lactose exceedingly difficult, or even preventing its crystallization in extreme instances.[1]

When acid is used as the precipitant for curd, as in making commercial casein, the resulting whey contains about 0.85 percent of protein and 0.75 percent of ash.[1] When rennet is used as precipitant, as in making Swiss or Cheddar cheese, the whey contains about 1.10 percent of protein and 0.50 percent of ash. The difference in percentage of ash is attributable to calcium phosphate, which is present in casein whey in greater quantities than in rennet whey. The greater percentage of protein in rennet whey is accounted for by the presence of a small proportion of acid-precipitable protein.

If casein whey is concentrated under vacuum, there is obtained a viscous syrupy mass from which lactose will not crystallize. If casein whey is neutralized, a precipitate forms, consisting of tricalcium phosphate and protein. The ratio of calcium phosphate to protein increases as the reaction of the whey approaches pH 7. If this "neutralization precipitate" and the remaining whey protein coagulable by heat are removed by filtration and the whey then concentrated, lactose will crystallize satisfactorily.

Sweet rennet whey, when adjusted to neutrality, does not form a precipitate, and it is possible to crystallize lactose from such a whey without previous coagulation of the whey protein. The presence of whey protein in the solution causes the concentrated whey to be viscous and filtration difficult, but this difficulty can be overcome either by concentrating the whey to a less degree or by obtaining large crystals by means of slow crystallization at a low temperature.[27] Yields of lactose are comparatively low when these procedures are followed, but it is practical to follow them if a food product of value is to be made from the filtrate. Whey protein may be removed from rennet whey by heating to boiling and acidifying, or it may be so finely dispersed by digestion with trypsin prior to concentration that contamination of the crystallized lactose is greatly decreased.

## MANUFACTURE OF LACTOSE FROM CASEIN WHEY

Until recently practically all of the lactose manufactured in the United States has been made from whey produced in the manufacture of casein by means of hydrochloric acid as precipitant. Whey containing sulfuric acid is not suitable because of the difficulty of removing slightly soluble sulfates that impart cloudiness to lactose solutions. The whey from casein precipitated by lactic acid, produced by bacterial fermentation of part of the lactose in the milk, is not used because of the relatively low percentage of lactose remaining in the whey.

The manufacture of lactose from casein whey consists of two distinct, though similar, operations; making of crude lactose from whey, and refining of crude lactose to produce lactose of U.S.P. grade (Figure 23). The refining operation may be applied to any crude lactose, however made.

**Manufacture of Crude Lactose.**[14, 21] Casein whey, which may contain 0.40 to 0.50 percent acid, calculated as lactic acid, is run to an iron tank and heated to boiling by means of live steam supplied through a perforated pipe set in the tank. During the heating, milk of lime is added gradually until the acidity is about 0.05 percent. Excess of lime should be avoided. It is not necessary to boil the whey, since coagulation of the whey protein will be complete at slightly below the boiling point. The steam is shut off and the whey allowed to stand for a few minutes to allow the coagulum of protein and calcium phosphate to settle as a sludge to the bottom of the tank.

The clear whey is drawn to a storage vat that feeds to a double- or triple-effect, vertical, vacuum evaporator, the sludge being left in the vat to be filtered later. The whey is concentrated to 20° Bé., which is equivalent to a content of lactose of about 30 percent. Foaming during evaporation can be minimized by having the evaporating pan clean at the start and by adding foam-reducing oils.

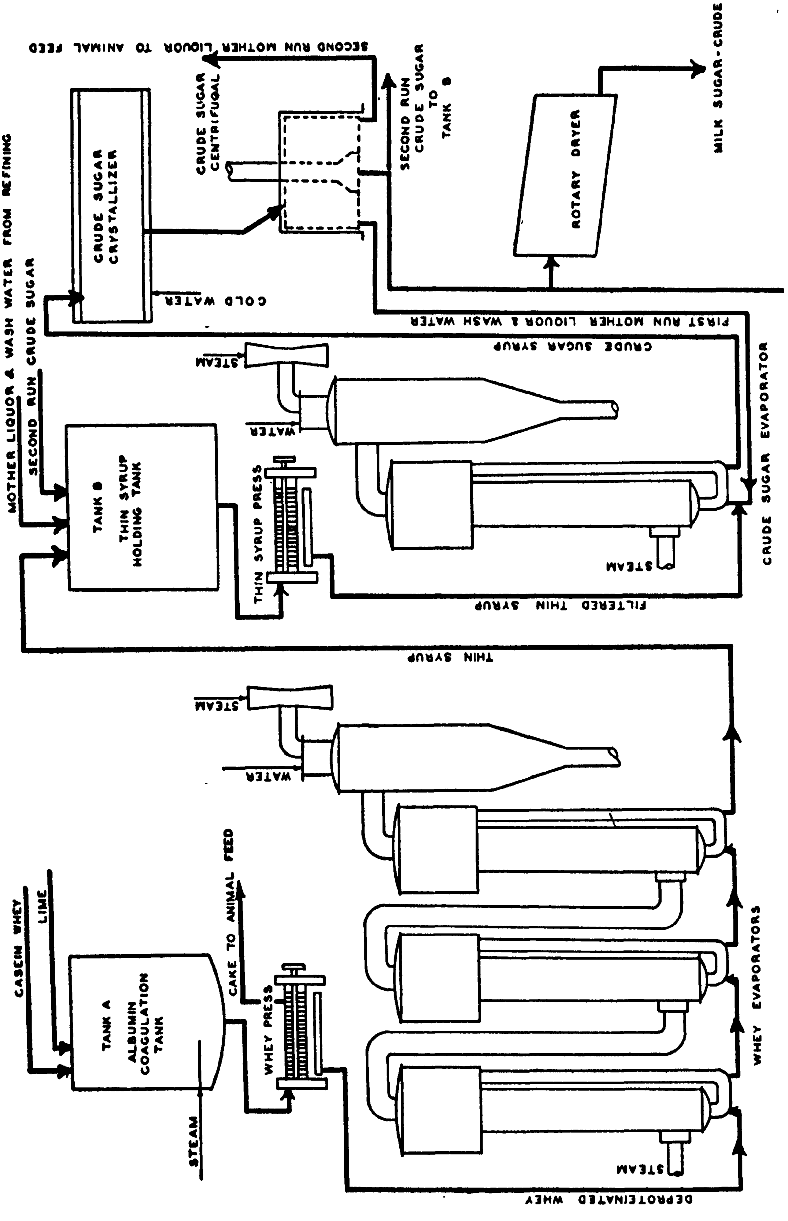
The protein and salts that separate during the first stage of concentration are removed by filtering the thin syrup through cloth in a filter press. As soon as the syrup is through the press, the sludge from the coagulating vat is run to the same press and the press cake blown dry. The whey derived from the sludge washes adhering syrup from the press. This wash whey may be combined with the filtered syrup or run to the vat for clarified whey. The press cake is suitable for use in feeds for poultry, and may be mixed with other residues from the manufacture of lactose, as will be described later.

The filtered syrup is drawn into a single-effect vacuum evaporator for the second stage of concentration. It is evaporated to 38 to 40°Bé., or the degree of concentration regulated by the judgment of the operator. The greater the degree of concentration, the greater the yield of sugar, but if evaporation is carried too far, the crystals will stick together in a solid mass, thus making filtering and washing difficult or even impossible. Part of the sugar crystallizes during the concentration in the pan, and the rest during the subsequent cooling in the crystallizing vat. The operator observes the progress of crystal formation by means of a sight glass, against which the crystallizing solution is thrown continuously by the boiling of the solution, and decides when the "graining" has progressed far enough and the "strike" should be made. Toward the end of the graining process some operators add a small quantity of hydrochloric acid, principally to diminish foaming, but also to prevent darkening of the sugar and precipitation of salts. One-half pint of 36 percent acid per each 1000 pounds of whey in the batch is a suitable amount.

The hot mass of crystals and syrup is dropped into crystallizing vats, which are rectangular tanks with round bottoms. These vats are provided with slow-speed worm agitators and with jackets in which cold water can be circulated. It is necessary to keep the crystals in motion to prevent them from "setting-up" into a solid mass. By suitable regulation of the speed of agitation and of the rate of cooling, uniform crystals of any desired size may be obtained. For most rapid crystallization and greatest yield, it is recommended that the crystallizing mass be cooled to about 85°F, held at this temperature for 3 hours, cooled to 70°F, and held at least 3 hours before filtering.

The soft, wet mass of crystals and mother liquor is fed by gravity to a basket-type centrifugal, in which the sugar is freed from mother liquor and washed. During the filtration and washing the basket should be spun at a rate of about 600 revolutions per minute. When mother liquor ceases running freely from the sugar, a small stream of cold water is played on the crystals from a hose until the mother liquor remaining in the sugar has been replaced by water. The speed of the basket is then increased to 1200 revolutions per minute in order to remove as much of the wash water as possible. The crude sugar is removed from the centrifugal and either refined promptly or dried immediately at 175°F in a tunnel drier.

The washings are run to the vat for clarified whey and the mother liquor


CASEIN WHEY
LIME
TANK A
ALBUMIN COAGULATION TANK
STEAM
WHEY PRESS
CAKE TO ANIMAL FEED
DEPROTEINATED WHEY
STEAM
WATER
STEAM
WHEY EVAPORATORS
THIN SYRUP
MOTHER LIQUOR & WASH WATER FROM REFINING
SECOND RUN CRUDE SUGAR
TANK B
THIN SYRUP HOLDING TANK
THIN SYRUP PRESS
FILTERED THIN SYRUP
STEAM
WATER
STEAM
CRUDE SUGAR EVAPORATOR
CRUDE SUGAR SYRUP
FIRST RUN MOTHER LIQUOR & WASH WATER
COLD WATER
CRUDE SUGAR CRYSTALLIZER
CRUDE SUGAR CENTRIFUGAL
SECOND RUN MOTHER LIQUOR TO ANIMAL FEED
SECOND RUN CRUDE SUGAR TO TANK B
ROTARY DRYER
MILK SUGAR-CRUDE

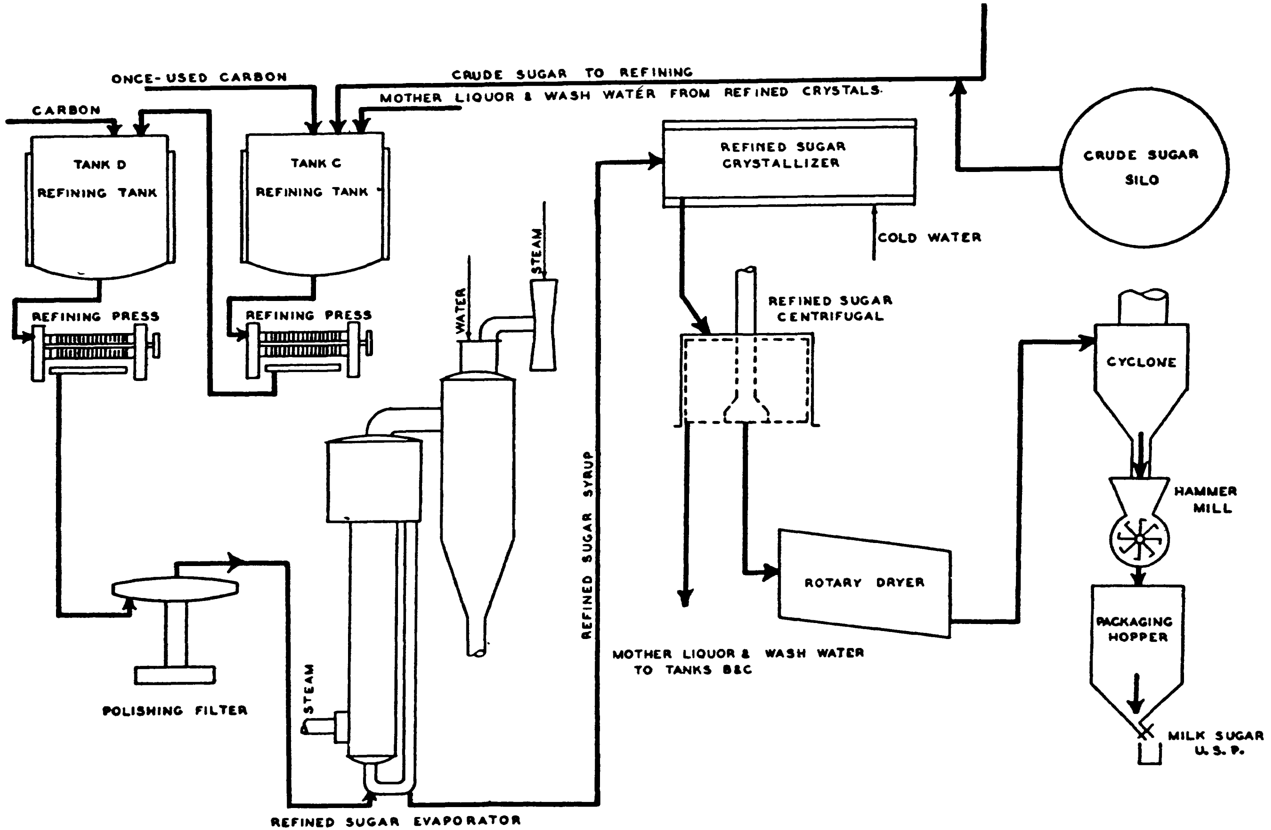


Fig. 23. Process for the manufacture of crude and refined lactose. (*Courtesy of Chemurgic Division, Sheffield Farms Company, Inc., Subsidiary of National Dairy Products Corporation.*)

is concentrated to obtain a second crop of crystals. The second mother liquor and washings are combined and used in making poultry feed as described below.

The first crop of crude lactose should comprise about 70 percent of the sugar present in the whey and contain between 85 and 90 percent lactose. It will be slightly yellowish in color. Because of the high viscosity of the second syrup, due to accumulation of salts, the second crop of crystals is difficult to separate and the yield is variable.

**Poultry Feed.**[21] The conversion of process residues into poultry feed is worthy of description, since the product is valuable and adds to the profit of the lactose operation.

The mother liquor and washings from the second crop of crystals are combined and concentrated in a vacuum evaporator to 36° Bé. This concentrated liquor and the whey protein obtained by filter-pressing the sludge obtained in the clarification of the whey are fed simultaneously into a horizontal cylindrical trough in which they are intimately mixed by means of a power-driven screw or worm. The components should be so proportioned that the mixture has a granular consistency and is not soggy. The material should be allowed to stand for a time to allow lactose to crystallize. It is then placed in an iron drum having a perforated bottom and, by means of a rotating agitator, is forced through the perforations and drops on screen-bottomed drying trays placed beneath. The filled trays are placed in a tunnel drier and the material dried at 160 to 180°F for 3 to 4 hours. The dried product is powdered in a hammer mill and packed in burlap bags provided with moisture-proof liners. The feed thus prepared will contain about 30 percent lactose and 25 percent protein.

**Refining of Lactose.**[14, 22] The refining of crude lactose to produce a sugar of U.S.P. grade comprises treatment of a solution of the lactose to remove color, protein and salts. Decolorizing carbon is used to adsorb color and probably removes other impurities to a slight extent; hydrochloric acid is added to facilitate the action of the carbon, to render salts soluble, and to aid in removal of protein; lime is used to adjust the reaction to that most favorable for the precipitation of protein and probably aids by combining with the protein to some extent. The carbon and the precipitated impurities are removed by filtration, the soluble impurities are largely removed in the mother liquor and washings from the centrifuged crystals.

The crude sugar is dissolved in water, or washings from a previous batch, in an iron tank by means of live steam, the proportions of sugar and water being adjusted so that the Bé reading is 20°, which indicates an approximately 30 percent lactose solution. For each 100 pounds of sugar in the batch, ¼ pound of a filter aid and 1 pound of decolorizing paste are added. The decolorizing paste consists of 3 parts of bone black, 1 part of activated carbon, 1 part of 36 percent hydrochloric acid, and enough water to make a paste easy to handle. The treated solution is boiled and its reaction adjusted to 0.09 percent acidity (calculated as lactic acid) by means of hydrochloric acid. At this point it is desirable that the solution stand over night in order that the decolorization process may reach its maximum effectiveness.

The following morning the solution is reheated nearly to the boiling point and milk of lime added cautiously until the acidity is reduced to 0.05 percent. An experienced operator can judge the proper reaction by observing the degree of flocculation, but it is recommended that accurate controls be used, since, if the solution is over-neutralized, the sugar will caramelize and become discolored. After the lime treatment, the solution is boiled vigorously for a few minutes and allowed to stand until the flocculated mixture of carbon, protein and insoluble salts has settled. It is then filtered, first through cloth in a filter press, and then through fine-textured rag paper supported between two perforated copper disks. Cloudiness in the filtrate indicates that insufficient lime was used; slow filtration or color in the solution indicates that too much lime was used.

The clear solution is drawn to a single-effect vacuum evaporator and concentrated to about 40° Bé. Hydrochloric acid in the proportion of about one-half pint to each 1000 pounds of whey represented in the batch is added to the last of the solution drawn into the pan to prevent precipitation of salts and protein in the concentrated syrup. The lactose is grained in the pan, crystallized, centrifuged, washed, and spun at high speed, as described for the making of crude sugar. The mother liquor is run to the vat for clarified 20° Bé. syrup for crude sugar production, and the wash water is used for dissolving subsequent batches of crude sugar for refining.

The sugar should be tested before it is removed from the centrifugal to make sure that it has been sufficiently washed. This is done by dissolving a small sample in distilled water and noting the color and clarity of the solution. Before drying, the lactose should be tested for protein, heavy metals, calcium, sulfates, and chlorides to determine whether the finished sugar will be of U.S.P. grade. Qualitative tests judged by an experienced technician are adequate for this purpose. If the tests indicate insufficient removal of impurities, the sugar may be marketed as technical lactose, put through the refining process again, or blended with a batch unusually low in impurities.

The damp lactose is spread on drying trays by hand and dried in a tunnel drier at a temperature of 180°F, the entering air being filtered through glass wool to remove dust and other possible contaminating substances. Drying is completed in two or three hours. The dried lactose is dumped into a hopper, provided with a cover to exclude dust and dirt, and fed to a pulverizer, in which it is ground until practically all will pass a No. 100 screen. The ground lactose is packed in 200- and 300-pound barrels, having liners of fine, unbleached muslin next to the sugar and water-proof paper liners between the muslin and the inner surfaces of the barrels.

The conversion of crude sugar to refined is about 90 percent, but the overall yield is nearer 100 percent, since the mother liquor and washings are reprocessed.

A sample of the finished sugar should be subjected to tests for purity to make sure that it meets the standards specified by the United States Pharmacopoeia.[15] It obviously is unnecessary for the manufacturer to test for impurities known not to be present, as, for example, starch. Some of the simpler tests can be made at the plant, such as those specifying that a solution of 1 gram of lactose in 20 cubic centimeters of distilled water shall be neutral to litmus, that a solution of 3 grams in 10 cubic centimeters of distilled water shall be clear, colorless and odorless, and that the sugar shall not contain more than 0.1 percent ash.

For a "chemically pure" lactose, it is considered reasonable[5] to require 99.7 percent purity as determined by the polariscope, not more than 0.020 percent nitrogen, not more than 0.020 percent fat, and not more than 0.050 percent ash. These are in addition to U.S.P. requirements.

For bacteriological use,[16] lactose must satisfy still other specifications. It should be free from ethyl alcohol by the iodoform test, and contain not more than 0.15 percent moisture. A 10 percent solution should show not more than a negligible turbidity when a solution either of silver nitrate or barium chloride is added. A 10 percent solution sterilized by heating for 30 minutes at 248°F should have a pH value not greater than 4.0, should remain acid on cooling, and should show no growth on incubation. Glucose should be absent as proven by the inability of *Bacterium typhosi* B to produce acid, or yeast to produce gas, in a sterilized solution of the lactose.

Certain users of lactose require that it shall contain no molds, as shown when tested by plating a 1 to 10 dilution in Sabouraud's dextrose agar, with $\frac{1}{10}$ milliliter of sterile 10 percent tartaric acid solution added to each 10 milliliters of medium, and incubating the plates at 90°F in a moist atmosphere for 5 days.

## MANUFACTURE OF LACTOSE FROM CHEESE WHEY

Seven procedures[27] are described below for isolating lactose from fluid cheese whey. The principal differences among these procedures may be seen in Table 40, which lists the principal operations of each and gives analytical data. The differences to be considered in making a choice among these processes are in simplicity and ease of operation, the condition of the recovered protein, the degree of purity of the sugar, and the yield. The theoretical yields given in the table for purposes of comparison should not be assumed to be actual yields; these will be 10 to 40 percent less than theoretical, depending on the efficiency of the operation. In many instances

TABLE 40. *Outline of Processes for the Manufacture of Lactose from Cheese Whey*

| | Process Designation | Condition of Whey Protein | First Clarification | First Concentration | | Second Clarification | Final Concentration | | Approx. Composition of Lactose (dry basis) | | | Theoretical Yield of Lactose per 100 lbs. Whey, lbs. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Total Solids, % | Sp.g. at 120°F, °Bé. | | Total Solids, % | Sp. g. at 120°F, °Bé. | Protein, % | Ash, % | Lactose, % | |
| 1 | Crude | Sol. | None | — | — | None | 57 | 30 | 2.8 | 2.4 | 95.3 | 4.0 |
| 2 | Improved Crude | Sol. | NaOH to change from pH 6.4 to 7.3 | — | — | None | 62 | 32 | 1.6 | 1.4 | 97.0 | 4.2 |
| 3 | Sweetened Condensed Whey | Sol. | None. Sucrose added equal to whey solids | — | — | None | 70 | 35 | 1.4 | 1.2 | 97.4 | 3.8 |
| 4 | Clarified Crude | Insol. | Heated to 100°F, HCl to pH 4.7 to 5.2. Clear whey syphoned | — | — | None | 64 | 34 | 1.5 | 1.5 | 97.0 | 4.3 |
| 5 | Technical | Insol. | Like 4, except whey filtered | 37 | 20 | Carbon added, boiled, limed to pH 6.3, boiled, filtered | 70 | 39 | 0.3 | 0.3 | 99.4 | 4.4 |
| 6 | Trypsin (Sweet Whey) | Insol. | Like 4, but after acid, boiled, limed to pH 6.3, boiled, filtered, digested with trypsin | 37 | 20 | Carbon added, boiled, settled, filtered | 70 | 39 | 0.1 | 0.2 | 99.7 | 4.4 |
| 7 | Trypsin (Sour Whey) | Insol. | Limed to pH 6.3, boiled, filtered, trypsin digested, carbon added, boiled, filtered | — | — | None, HCl added to whey in pan | 65 | 35 | 0.1 | 0.1 | 99.8 | .3 |

it may not be advisable to try for high yields; it is usually easier and often more profitable to leave the rest in the mother liquor for diversion to food products or feed.

**Crude Process.** Separated, sweet, cheese whey is concentrated in a vacuum evaporator to 55 to 60 percent solids (30° Bé.) and dropped to a crystallizing vat. The crystals of lactose should be allowed to grow slowly at a low temperature with only occasional agitation. This will give large crystals, avoid overheating during crystallization and make the separation of the crystals easier. The lactose is centrifuged, washed with cold water, spun at high speed to remove the final wash water, and dried in a tunnel drier at 175°F. If the concentrate is slimy and the crystals are small, there will be filtration difficulties, which can be lessened by condensing to less solids content. The product of this process is highly contaminated with protein and salts. The wet cake from the centrifugal may vary widely in composition, but generally will contain approximately 86 percent of lactose, 5 percent of protein plus ash, and 9 percent of water. The whey protein in the mother liquor is in soluble condition.

**Improved Crude Process.**[2] Sweet whey having approximately 0.2 percent acidity is neutralized with sodium hydroxide to 0.04 percent acidity (pH 7.3). The neutralized whey is warmed to 140°F and condensed to about 62 percent solids (32° Bé). The concentrate is cooled to between 32 and 38°F and held for at least 18 hours with occasional stirring. The lactose is centrifuged, washed, and dried as in the Crude Process. The crude sugar prior to drying will contain approximately 91 percent lactose, 3 percent protein plus ash, and 6 percent water. The soluble whey protein is suitable for use in food or feed.

**Sweetened Condensed Whey Process.**[17, 26] The manufacture of sweetened condensed whey is described in Chapter 4. For recovery of lactose, which is a secondary product of the process, the concentrate should be cooled to room temperature or lower, seeded with a few crystals of lactose, and allowed to stand over night. Stirring several times will accelerate crystallization, but cause the crystals to be smaller than if the concentrate is not stirred. The use of lower temperatures will increase the yield, which is only 1 to 1½ pounds per 100 pounds of whey. The crystals are centrifuged, washed, and dried. The crude lactose contains a trace of sucrose and gives a turbid solution in water.

**Clarified Crude Process.** The whey is heated to boiling and clarified in either of two ways: it is acidified by means of hydrochloric acid to a reaction between pH 5.0 and 5.2; or, calcium chloride in the proportion of 3 pounds to each 1000 pounds of hot whey is added and followed by milk of lime sufficient to bring the reaction to approximately pH 6.0. The flocculated protein is allowed to settle, and the clear whey is syphoned to the evaporator and concentrated to about 64 percent solids (34° Bé.). Crystallization, centrifuging, washing and drying are conducted as in the Crude Process. The composition of the crude sugar is approximately the same as that from the Improved Crude Process, but the yield based on the quantity of whey drawn into the evaporator may be slightly greater. The whey protein in the sludge is insoluble and may be utilized in feed. Insoluble whey protein is considered less desirable than soluble whey protein for use in foods.

**Technical Process.** The whey protein is coagulated as in the Clarified Crude Process, by treatment at the boiling point with either hydrochloric acid or calcium chloride and milk of lime. The clarified whey is syphoned from the sludge, or, preferably, filtered with filter-aid added to expedite the operation, and evaporated to about 37 percent solids, equivalent to 30 percent lactose or 20° Bé. This syrup is heated to boiling, treated with decolorizing carbon and lime, evaporated, grained, crystallized, washed and dried, all as described previously under the subject of refining of lactose. The product may give a slightly turbid solution in water, and will contain about 0.3 percent ash and 0.3 percent protein. It is suitable for use in foods and for other purposes for which the U.S.P. grade is not required.

**Trypsin Process with Sweet Whey.** This process consists of the Technical Process with the addition of a trypsin treatment to decrease the percentage of protein in the sugar. The trypsin hydrolyzes the residual whey protein sufficiently so that it is largely removed in the mother liquor from the centrifuging of the sugar. In the first clarification treatment the pH value of the boiled whey is adjusted to pH 6.3 by means of lime. The whey is boiled again, then filtered and run to a vat in which the temperature can be maintained at 132 to 138°F. One pound of trypsin is added for each 10,000 pounds of whey and the temperature held between 132 and 138°F for an hour, at the end of which time the digestion should be complete, as shown by the trichloroacetic acid test. The treated whey is then evaporated to 20° Bé. and further processed as described for refining of lactose. The product should contain not more than about 0.1 percent of protein and should give a clear water solution that should foam only slightly on boiling.

The trichloroacetic acid test for completeness of digestion consists of adding 2 milliliters of a 40 percent solution of trichloroacetic acid to 2 milliliters of the whey in a test tube. Undigested whey gives a heavy white precipitate, a sufficiently digested whey only a trace of turbidity.

The foaming test consists of weighing 10 grams of the finished lactose into a 250 milliliter beaker, adding 15 milliliters of water and quickly heating the solution to boiling. The amount of foam produced during vigorous boiling is an index to the purity of the sugar. A solution of a sugar containing more than 0.05 percent protein will foam; if the percentage of protein is greater than 0.2 percent, the solution will boil over the top of the beaker.

**Trypsin Process with Sour Whey.** If sour whey is to be used for making a technical grade of lactose, the trypsin process can be shortened by applying the carbon treatment to the whey immediately after the tryptic digestion and completing the evaporation in one step. It is desirable to add toward the end of the evaporation ½ pint of hydrochloric acid for each 1000 pounds of whey being evaporated. The trypsin digestion may be omitted if low protein content and non-foaming characteristics are not of importance. This procedure is not suitable for making lactose from sweet whey, because a heavy precipitate, consisting largely of calcium phosphate, forms during the condensation of such a whey.

**Alcohol Process with Dried Whey.**[12, 13] This procedure produces three products,—impure whey protein, relatively pure lactose, and a riboflavin concentrate. The method used is based on the facts that the protein is insoluble in ethyl alcohol, and that amorphous lactose will disperse in ethyl alcohol, forming a supersaturated solution from which the lactose will subsequently crystallize.

The whey should have been spray-dried and should be freshly made or should have been stored in a cool, dry place, since the success of the operation depends on the lactose being in the amorphous, or glassy, state. The dried whey is stirred rapidly into 15 times its weight of 76 percent (by volume) ethyl alcohol, and, after 3 minutes, the insoluble protein is removed by filtering through cloth in a filter press. The filtrate is acidified to a reaction of about pH 3.6 and placed in a refrigerator over night to crystallize. The lactose is then removed by means of a sugar centrifugal. A white sugar of a high degree of purity is obtained, the yield being 75 percent of the lactose in the dried whey, or 80 percent of that in the filtrate from the filter press. If 95 percent alcohol is used instead of 76 percent, the yield is less and the lactose is in the form of the equilibrium mixture of alpha and beta rather than the usual alpha hydrate. A vacuum shelf drier and an alcohol still are necessary to recover the alcohol from the products of the operation.

## MANUFACTURE OF BETA LACTOSE

Because beta lactose is initially more soluble in water than is alpha lactose, there is a demand for beta lactose that has been met by the devising of several processes for the conversion of alpha lactose, either wholly or in part, to the beta form. The principles underlying these procedures are discussed in Chapter 1. The processes described briefly here are protected in most instances by patents, which should be consulted before attempting to produce beta lactose commercially.

Drying lactose solutions by the spray process produces a mixture of amorphous alpha and beta lactose in approximately the equilibrium ratio.[3] Initially this mixture is more soluble than alpha lactose, but less soluble than a mixture of the two forms in the ratio of their individual solubilities. It has a high rate of solution because of its amorphous condition, but is hygroscopic and has poor wetting properties.

By drying lactose solutions on a drum or roller drier, a sugar having 90 to 99 percent in the beta form can be produced.[3] The product is somewhat less amorphous in appearance than spray-dried lactose, is much less hygroscopic, and has relatively good wetting properties. Lactose containing more than 90 percent of beta has been produced by drying a preheated 80 percent solution of lactose on an atmospheric drum drier by using a steam pressure of 65 to 75 pounds and a drum speed of 5 to 7 revolutions per minute. Increasing the drum speed beyond a certain rate decreases the proportion of beta lactose in the dried product. The speed and temperature to be used with any specific drying unit should be determined experimentally on that unit.

The process of Supplee and Flanigan[23] consists of drying a lactose solution in the form of a film on a heated surface at a temperature above 212°F and removing the film from the surface while it is still a paste containing at least 2 percent water. The lactose crystallizes in the beta form and the heat remaining in the paste completes the drying.

The first process of Sharp[18] consists of alternately adding alpha lactose to a solution saturated with lactose and maintained at a temperature above 201°F and removing an equivalent quantity of lactose crystallized in the beta form. Since a saturated solution of lactose above 201°F is saturated with respect to beta, but not with respect to alpha lactose, more of the alpha form can be dissolved, but, since equilibrium between the two forms in solution is established almost instantaneously, at this temperature, the solution immediately becomes supersaturated with respect to beta lactose and this form crystallizes from the solution. By filtration in a heated centrifugal, a sugar containing a high percentage of beta lactose is obtained.

More recently, Sharp and Hand[19] have developed a simpler procedure in which dry alpha lactose hydrate is heated in a closed container under pressure to 248 to 266°F. In the presence of the water vapor formed, a solution of alpha lactose is produced on the surface of the crystals and beta lactose crystallizes from this solution. When the conversion has proceeded to the degree desired, the pressure is released and the water vapor is allowed to escape.

The method of Verschuur[8, 24] has not been tried, apparently, on a large scale, but is described here because of the claim made that the beta lactose contains no alpha and because of the interesting procedure employed. Pyridine is added to a boiling solution of lactose in water. The pyridine and water are distilled together, more pyridine being added from time to time. When the proportion of water becomes sufficiently small, beta lactose crystallizes from the solution. The crystals are washed with boiling pyridine, then with hot alcohol, and finally dried.

## HYDROLYZED LACTOSE

The hydrolysis of lactose has been studied by Ramsdell and Webb[17a] and a procedure developed for the preparation of a hydrolyzed lactose sirup suitable for use in foods. It is necessary to use a good grade of lactose, since, if whey or crude lactose is used, the product contains caramel and charred material. A charge of 2100 grams of lactose, 49 grams of normal hydrochloric acid and sufficient water to give a total weight of 7000 grams is introduced into a 3-gallon, glass-lined, steam-jacketed pressure kettle. Seventy pounds of steam pressure is applied for a period of 65 minutes. After the first five minutes the temperature should be 297°F. The charge is stirred at the beginning of the heating until all the lactose is dissolved. After the heating, the mixture is treated with decolorizing carbon, filtered, and condensed under vacuum to 60 percent solids. Sodium bicarbonate is then added, with stirring, until the reaction is pH 4.9–5.0.

Sirups prepared by this method have contained approximately 26 percent glucose, 29 percent galactose and 4 to 5 percent of unidentified byproducts. There is a tendency for color to develop, especially at more alkaline reactions above pH 6.0. Since the solution is supersaturated with the sugars, some galactose occasionally crystallizes on storage. The sirup appears to be satisfactory as a food sweetener or as a table sirup.

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